GULF CANADA LIMITED

RESEARCH AND DEVELOPMENT DEPARTMENT

SHERIDAN PARK, ONTARIO, CANADA

THE BENEFICIATION OF LOW RANK CANADIAN COALS WITH HEAVY RESIDUA (PHASE II)

Final Report

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1. SUMMARY

The main objective of the Phase II program was to develop the batch coal upgrading process with heavy residuum into continuous operation. Two continuous processing units were constructed and the process technology has been extensively studied. Results of the experimental program showed that it was possible to upgrade low rank coals with this process for a short period of operation (1 hour), when coal particles are coarser than 3.2 mm. Products obtained during this period also showed improved qualities and handling characteristics. However, it became difficult or even impossible to control the residuum level on upgraded products in an extended period of operation. An alternative processing method to solve this problem is recommended for future development.

The results of the Phase II beneficiation program are summarized below:

1.1 It has been demonstrated in a pilot plant scale that preparation of high softening point residuum by mild thermal treatment of VTB from conventional crude presents no operating problems. Suitable residuum, therefore, can be made available from commercial operation in addition to residua from heavy oil operations.

1.2 A batch reactor (20-litre) and two laboratory scale continuous units (Rotary Kiln Reactor and Stirred Tank Reactor - Derrick Screen System) were designed and constructed for this research program.

1.3 The batch experimental program confirms that large coal particles (38.1 mm X 12.7 mm) can be effectively dewatered under similar experimental conditions used for smaller particle sizes (12.7 mm X 2.0 mm). Immersion temperature and residence do not have critical effects on dewatering within the range under study.

Product coals from large particle sizes typically show 70-95% moisture removal with 20 and 45% increases in heating values for subbituminous coal and lignite, respectively.

1.4 Continuous processing of coal in the rotary kiln reactor, which has the advantage of combining coal immersion and draining of excess residuum in a single unit, gives serious operational problems. The rotary kiln reactor acts as a size classifier which conveys coarse particles but leaves fine particles in the residuum pool. It also suffers from serious limitations of heat transfer, resulting in a reduced capacity for coal feed an order of magnitude lower than the designed flowrate. Finally, this reactor fails to recover itself from process upset. These factors this prohibit any further investigation of this processing scheme.

1.5 Continuous beneficiation of coal in a two-stage processing scheme, a stirred tank reactor followed by a vibrating screen, also shows difficulty in delivering consistent products of residuum level within economic limits. Each single stage of this processing method is performing quite satisfactorily; but an integration of both steps can only give satisfactory performance for a relatively short period of time (1 hour). The exact reason for this is, however, not clear, although chemical bondings between particles and residua are suspected.

1.6 Both processing schemes studied in this research program required a step of draining excess residuum. This step has greatly complicated the operation of the continuous unit. If this step can be avoided by an alternative process, then the chance of success for a continuous operation will be greatly enhanced. The elimination of the draining step should be emphasized in any future research direction.

2. INTRODUCTION

This report describes work performed during 1984-85 on D.S.S. Contract Serial No. OSQ84-0097, "Beneficiation of Low Rank Canadian Coals with Heavy Residua - Phase II". The contract originally consisted of 13 tasks (Appendix 1); but the work statement was revised due to operating problems encountered in the first continuous unit designed and built for this contract. A progress report (Appendix 2) was submitted to the Scientific Authority in July 1985 and an amendment of the work statement (Appendix 3) was made to accommodate the completion of the key components and objective of this research program.

The primary purpose of this research program is to develop this beneficiation method in a continuous mode laboratory scale operation. Other specific objectives of this contract are:

- To prepare and analyze heavy residuum (Tasks 1 to 3)
- To prepare and analyze coal samples (Task 4)
- To carry out a batch experimental program for large coal particles (38.1 mm X 12.7 mm) (Tasks 5R and 6R)
- To design, construct and commission a continuous laboratory unit and to carry out experiments in this unit (Tasks 7, 8, 9R, and 10R)
- To assess experimental results and to comment on process economics (Tasks 11R and 12R)

Activity and progress in all tasks, except one, are discussed in detail in this final report.

However, the study of process economics was not performed because the immersion technology in a continuous operation was not entirely successful.

3. ANALYSIS OF COAL SAMPLES

This research program continued to use Wabamun subbituminous coal and Poplar River lignite which were studied in D.S.S. Contract No. OSQ83-0053 (Phase I Study). In this program the top size of the coal samples was extended to 38.1 mm with the objective of expanding the applicability of this upgrading process. Top size of coal samples used in the Phase I study was 12.7 mm.

Wabamun subbituminous coal (95 kg) and Poplar River lignite (140 kg) were individually screened to obtain the 38.1 X 25.4 mm and 25.4 mm X 12.7 mm size fractions by Witteck Development Inc. in Mississauga. These size fractions were subsequently analyzed by Loring Laboratories Ltd. in Calgary. The results of proximate, ultimate analyses and heating values of these coal samples are presented in Table 1.

Wabamun and Poplar River coals have residual moistures of 12.5% and 24.5%, respectively. These samples are ideal feedstocks for studying the dewatering process.

4. PREPARATION AND ANALYSIS OF HEAVY RESIDUUM

The Vacuum Tower Bottoms (VTB) from Gulf's Edmonton Refinery was selected for this research program. Since Edmonton VTB has too low a softening point (45°C) to be used for coal beneficiation. Thermal treatment (504°C+ or higher cut point fraction after thermal cracking) had been carried out to increase the softening point.

Based on thermal cracking data generated from autoclave study in the Phase I Study, the thermal treatment of 5 barrels of Edmonton VTB was carried out in Gulf's Donor Refined Bitumen pilot plant (1 bbl/d) at Sheridan Park. Thermal treatment conditions were 420-425°C and 43-50 min residence time. The reactor effluent was distilled to 360°C in the vacuum tower in the DRB unit. The unit ran for a total period of 8 days.

The 360°C+ fraction was further distilled in an Equilibrium Flash Vaporization (EFV) unit to generate pitch. The cut point was adjusted to produce a pitch of 95°C softening point (62.3 wt% of VTB feed). This pitch was used as the immersion medium for this research program.

The operating condition, and product yields of pilot plant thermal cracking are summarized in Table 2. The properties of the pitch are presented in Table 3.

5. BATCH EXPERIMENTAL PROGRAM

In order to study the beneficiation of large coal particles (38.1 mm X 12.7 mm) and minimize sampling errors, a large reaction vessel of 20-litre capacity (MK-II Reactor) was designed and constructed for the batch experimental program. This reaction vessel is basically similar to the one used in D.S.S. Contract No. OSQ83-00053 (Phase I) but is equipped with a stirring mechanism to ensure a uniform temperature of the residuum in the reactor. Photographs of the MK-II reactor are shown in Appendix 4.

The objective of this batch experimental program is to expand the applicability of this upgrading method to larger coal particle sizes. Since removal of residual moisture from larger coal particles is suspected to be more difficult than from smaller sizes, the degrees of dewatering were given particular emphasis in this program. Consequently, experimental conditions at high temperature, short residence time and low temperature, long residence time were used in this study. These conditions therefore should not be interpreted as the optimum conditions for the products.

The results of this batch experimental program for Wabamun subbituminous coal and Poplar River lignite are summarized in Table 4 and Table 5, respectively. These data show that dewatering from larger coal particles does not present any special problem under the conditions studied in this program. A brief comment on these results are given below.

5.1 Water Reduction

Water removals for Wabamun subbituminous coal and Poplar River lignite of particle sizes 38.1 X 12.7 mm are 70-95% and 75-87%, respectively. There are no definite trends for the degrees of water removal when they are related to immersion temperatures. A minimum removal of 70% of the residual moisture in the coal samples was observed under similar experimental conditions used for smaller particle sizes.

5.2 Heating Values

Average increases in heating values for Wabamun subbituminous coal and Poplar River lignite are 20% and 45%, respectively. These increases, again, are consistent with previous observations on smaller particle sizes.

5.3 Residuum Content

Residuum contents for Wabamun coal range from ca. 4% to 7% for particles 38.1 mm x 12.7 mm under draining temperatures between 180°C to 205°C. No definite trends can be found between residuum contents and draining temperatures. Residuum contents for Poplar River lignite vary between 11% to 29%, and draining temperatures showed a definite effect on residuum content. Higher draining temperature gives a product of lower residuum content. The differences in residuum content between lignite and subbituminous coal again suggest that the high level of residuum in lignite is due to penetration of residuum into coal pores which are more abundant in lignite.

5.4 Other Properties

Ash content, volatile matter content, and sulfur content increase as a result of dewatering and addition of petroleum residuum. The large variations in ash content also show the sampling problems, despite the fact that large quantities of samples were used in these experiments.

6. CONTINUOUS UNIT EXPERIMENTAL PROGRAM

The design, construction, commissioning, operation, and evaluation of products from the laboratory-scale continuous unit are discussed in this section.

6.1 Design

6.1.1 Process Description

In this process low rank coals are immersed in hot petroleum residua. Moisture and some labile oxygen are driven out from the coals. Excess residua are then allowed to drain off from the product. The calorific value of the coal is increased by dewatering and surface coating and internal absorption of the residuum. This residuum protects the coal product from moisture readsorption and therefore reduces the tendency of the coal to spontaneous combustion.

6.1.2 Process Variables

Five process variables, which affect coal beneficiation, have been studied in a batch operation in Phase I study. These variables are coal quality, residuum quality, coal particle size, temperature, and residence time.

In general, it can be said that the final coal product properties and yield will depend mainly on the properties of the coal feedstock and the coal particle size. The operating temperature will also affect the final product yield, while the residuum quality will affect the final product properties significantly. On the other hand, the residence time and residuum quality were found to influence the product yields only marginally, and the same applied to the temperature and residence time effect on the final product quality.

6.1.3 Design Basis

Typical operating conditions and range of operating conditions for the continuous unit are as follows:

	Typical	Range
Temperature	240°C	200-280°C
Residence time	10 min	5-20 min
Particle size	38.1 mm X 3.2 mm	38.1 mm X 3.2 mm
Residuum consumption	6% wt of Coal Feed	3-10% wt
Product yield, % wt	90	85-95
Water recovered, % wt*	8	6-10
Gas Yield, % wt	1/2	1/2-1

* Does not include surface water.

6.1.4 Design Philosophy and Description of Selected Schemes

The design philosophy of the laboratory scale continuous unit is to use equipment that is proven at high capacities. This will allow easy scale-up in the future. Of the many options examined for continuous coal beneficiation, rotary kiln and stirred tank reactor are considered more favourable than other devices such as auger, drag classifier, bucket wheel, etc. Rotary kiln was selected because of the possibility of immersion and draining in one single unit, and the stirred tank reactor followed by solid-liquid separation was to be used as a back-up system.

A flowsheet showing the laboratory scale continuous unit based on rotary kiln is presented in Figure 1. In this design, coal is fed into a rotating helicoidal conveyor, the reactor, which is partly filled with hot residuum. The hot residuum drives the moisture out of the coal and is absorbed into the voids of the coal, also coating the surface of the coal particles. The coal is conveyed along the rotary drum which is tilted at an angle of 5° to 15° from horizontal. It then emerges from the rotating conveyer and is immediately cooled in water. Photographs of this continuous laboratory unit are given in Appendix 5.

The flowsheet of the two-stage process, a stirred tank reactor followed by vibrating screens, is presented in Figure 2. In this design, coal is continuously fed into a reactor for drying. After a specific residence time, treated coal overflows onto a vibrating screen where excess residuum can be removed. Residuum separated

from the screen is recycled back to the reactor. Photographs in Appendix 6 depict the components of this design.

6.2 Construction

The two continuous laboratory units were designed by the Engineering Group at Gulf Canada's Research and Development Department. Procurement of equipment and construction of the units were also carried out in-house. While two different reactors were utilized in the continuous processing of coal, the following are common components which are required for the operation of these units:

- Coal Feeder (5-50 kg/h)
- Residuum Feeder
- Residuum Level Control System
- Temperature Monitoring System
- Gas Burner for Process Heat (60 000-800 000 BTU/h)
- Ventilation System
- Operation Console

The rotary kiln unit was built in the Spring of 1985 and the stirred tank reactor was assembled in the Summer of 1985. All the design drawings for these units are attached.

6.3 Commissioning

This section discusses the commissioning of the two laboratory-scale continuous units, designed and constructed for this research program. The discussions will concentrate only on the effort spent in commissioning these units and the rationale behind these activities. Calibration of unit components are considered as "routine" procedures in the operations of new systems, and will not be mentioned in this report.

6.3.1 Rotary Kiln Reactor

6.3.1.1 Preliminary Runs

The objective of the preliminary runs were to commission the rotary kiln reactor for continuous coal beneficiation.

The test conditions used for these experiments were:

Kiln rotation speed:	0.3 rpm
Kiln angle:	4.5°
Temp. of Pitch:	235°C
Temp. of Gas at Inlet:	350°C
Coal feed rate:	2.5 kg/h
Coal particle size:	9.6 X 3.2 mm (Test 1)
	6.4 X 3.2 mm (Test 2)

The results of these preliminary runs are summarized below:

Test 1 Operation continued for 1.5 hours with acceptable products. After that residuum was conveyed to the top of the kiln and emerged with products. Operation was forced to shut down.

Test 2 Products did not emerge from the kiln after operation for 1 hour. Test run was stopped and it was observed that fine coals were stuck with pitch to kiln walls and conveyor flights inside the kiln.

In both tests, coal feed rate had to be kept at 2.5 kg/h to maintain the temperature of pitch at 235°C. With the rotary kiln operated at 0.3 rpm, feed rate of coal should be in the order of 20 kg/h. This suggested serious limitations in heat transfer. Questions arising from these preliminary runs are:

1. Was the fine coal present or generated in the operation responsible for poor solid-liquid separation?
2. Was there any change in residuum viscosity due to heat treatment and/or mixing with coal fines that made separation difficult?

A series of model experiments were carried out to answer these questions.

6.3.1.2 Model Experiments

A series of model experiments using light oil and stone aggregates were carried out with the objectives to answer the questions arisen from the preliminary test runs, and to better understand the separation mechanisms inside the rotary kiln. A light oil with viscosity at room temperature similar to that of the pitch was chosen because operation at ambient temperature will not change the rheological properties of the immersion medium; and stone aggregates

were used to eliminate the possibility of fines generation from the operation.

Test Conditions were:

Kiln rotation speed:	0.3-1.2 rpm
Kiln angle:	4.5°
Temp. of light oil:	23°C
Temp. of gas at inlet:	23°C
Aggregate particle sizes:	A - 19.0 mm X 12.7 mm (test 1)
	B - 9.6 mm X 6.4 mm (test 2)
	C - 6.35 mm X 3.2 mm (test 3)
	D - 4.0 mm X 3.2 mm (test 4)
	E - 3.2 mm X 0 (test 5)
	F - 10% A + 20% B + 30% C + 40% E (test 6)

Similar results were obtained from all test conditions. The following were observed:

1) The drain holes in the flights did not block with any size of stone.
2) Only a small portion of the free oil in each flight returned through the drain holes, with the major portion bypassing the holes and exiting at the top of the kiln.
3) All the oil which did pass through a drain hole flowed into the stone layer of the following flight.
4) A larger amount of oil was trapped between the stone and the kiln wall and was dragged out the top of the kiln.
5) Stone of approximately 3.2 mm stuck to the kiln walls and flights and did not move up the kiln.
6) Fine material accumulated in the pitch pool and did not move up the kiln.

The results of these tests showed that the rotary kiln reactor was operating as a size classifier, with the end result that fine particles were not conveyed to the exit (Fig. 1). This classifying action was observed with light oil at room temperature which causes no change of viscosity. It was apparent that the rotary kiln reactor was not suitable to continuously process coal based on the

observation that it was not possible to drain off the excess light oil from the upgraded product.

6.3.1.3 Recovery Test of Rotary Kiln Reactor

The model experiments showed some serious design and operating problems when the rotary kiln reactor was utilized for continuous coal beneficiation. While major equipment modifications such as installation of a concentric drum screen, or caps over the initial flights to prevent solid recycling may offer some improvement in the operation, these changes are not considered practical if the rotary kiln reactor cannot satisfy a recovery test.

A recovery test is a simple but critical evaluation of commercial equipment when they encounter process upset. A processing unit must be able to recover from the upset by normal operation. In this case, when coal fines are stuck with pitch to the kiln walls and flights, these materials must drain back to the pitch pool from this upset by simple heating without the aid of any mechanical clean-up. It was observed that the rotary kiln built cannot satisfy this critical evaluation.

The failure of the rotary kiln reactor indicates the need to evaluate other methods for continuous operation.

Work carried out at Gulf under a separate task led to the development of a two-stage processing system consisting of a stirred tank reactor-screen separator. This system is described and used in the following experiments.

6.3.2 Stirred Tank Reactor - Screen Separator

The stirred tank reactor system for continuous beneficiation of coals was initiated after the failure of the rotary kiln reactor. Stirred tank reactors, like the rotary kiln reactors, are mechanically proven at commercial scale. In addition, the rather low coal to residuum ratio in a stirred tank reactor operation should eliminate the heat transfer problems observed in the commissioning of the rotary kiln reactor.

The stirred tank reactor, however, requires a second stage process for draining residuum for the delivery of product coals. This

necessitated the evaluation of solid-liquid separation processes. A comprehensive evaluation program on pitch characteristic changes due to heat treatment and solid-liquid separation was carried out before the actual design and construction of a second unit for continuous coal processing.

6.3.2.1 Pitch Characterization Tests

A program to determine if there is any change in pitch properties was carried out under a simulated condition of a stirred tank reactor. Steam was injected into a stirred tank reactor and a pitch sample taken every hour and analyzed for softening point and viscosity. These tests were also performed on clean pitch and pitch containing 20 wt% of coal of 1.00 mm X 0 particle sizes. Changes in pitch qualities will have an important effect on residuum draining by screening and consequently the operability of the process and product qualities.

The data of pitch characterization are presented in Table 6. These tests were conducted with about 15 kg of pitch in the beginning of the run, and about 1.4 kg of steam/h injected into the reactor at 210-240°C. 500 g of sample was taken out every hour and analyzed. The results of these tests show that there is little change in both viscosity and softening point of the pitch. These observations, therefore, indicate that the first stage processing is technically feasible.

6.3.2.2 Pitch Draining Tests

The second stage processing in a stirred tank reactor system is solid-liquid separation which controls final residuum content. While centrifuges and filters are anticipated to generate products with lower residuum contents, the use of this equipment, however, will significantly increase the complexity of coal beneficiation. Screening, either stationary or vibrating, would be more practical and simple to operate, if they can deliver products with acceptable residuum contents. A series of tests were carried out on screening with this objective.

Stationary Screens

The first attempt was to use stationary screens for solid liquid separation. In these tests, screens with long slot wedge and round hole openings were examined. Initial experiments with Paraflex oil, which has similar viscosity as pitch at 240°C, showed that experiments with hot pitch was necessary as surface tension may also be an important factor in determining the final residuum content of the product.

The results of the stationary screening tests with Paraflex oil are presented in Table 7. Particle sizes of coal used were 3.2 mm X 1.0 mm, and the openings were 1 mm for both types of screens. It was observed that for experiments using light oil screens of either type were blinded by small particles and coal particles had high retentions of oils.

The results of the stationary screen tests with hot pitch at 240°C are also presented in Table 7. Despite the fact that pitch at temperature has similar viscosity as the Paraflex oil at room temperature, the residuum contents of the products were greatly reduced. Although the time required for draining to produce acceptable residuum content was long, this indicates that the use of screen as a second stage processing for the stirred tank reactor is technically feasible.

Vibrating Screen Tests

In order to increase the efficiency of separation by screening, vibrating screens were examined to determine their technical merits. Both the Sweco vibrating screen and the Derrick Flo-liner cleaner were investigated.

Sweco vibrating screen takes coal in the center of a circular screen and discharges solid around the circumference. It has a combined vertical and circular motions in separating solids from liquids. The results of tests using a 46 cm diameter screen for a 20 wt% coal in pitch (12.7 mm X 9.5 mm) are summarized in Table 8. These results show that acceptable residuum contents on the products can be expected at various residence time and various openings of the screen. These data also indicate that evaluation

of vibrating screens with other mechanism and proven commercial applications should be carried out.

The Derrick Flo-liner cleaner represents a logical extension of this effort. Derrick screens use an elliptical vibrating action which flows particles forward along the length of the screen with 3 to 6X G forces. An 46 cm wide X 244 cm long Derrick screen was purchased for this purpose and was installed to accept the overflow from the stirred tank reactor. A series of tests were carried out to establish operating conditions and verify the suitability of this unit. The results of these tests were tabulated in Table 9. These data indicate that the Derrick screen produces satisfactory products when slurries of 20 wt% solids of particle size 9.5 X 6.4 mm were used.

6.4 Experimental Program on Continuous Laboratory Unit

Based on the results of pitch characterization and Derrick vibrating screen, the continuous laboratory scale unit was assembled with the stirred tank as the first stage and Derrick screen as the second stage. The feasibility of continuous processing coals by this unit was investigated.

Size fractions of 25.4 mm X 6.4 mm of both Wabamun subbituminous coal and Poplar River lignites were used in these experiments. Feed rates of coal were set at 7.5 kg/h and the operating conditions for these experiments are presented in Table 10.

Both experiments ran well for about one hour, but a mat of coal particles started to form on the Derrick screen as the experiment continued. For the Wabamun coal, the mat appeared relatively dry; but for the Poplar River lignite, a great deal of residuum was trapped with coal particles which were also much finer in terms of particle size. These mats stopped products from coming out the exit and the operations were forced to shut down.

This observation was confirmed when some other coals were processed through this unit. While a more academic approach to investigate the bondings between coal and residuum will probably be able to narrow down the reasons why the two-stage approach does not perform, Gulf Canada is of the opinion that a better understanding of

the problem is unlikely to improve the commercialization of this process. The experimental program was terminated. Effort is being continued to find an alternative method for the coal upgrading process with heavy residuum.

6.5 Product Evaluation

Despite the fact that these continuous operations were not entirely successful, the products generated in the first hour were collected and analyzed to gain some insight of the product quality. The qualities and properties of the products are compared with their parent coals in Tables 11 and 12 respectively. They are briefly discussed below:

Water - About 90% of the residual moisture was removed for both coals.

Ash, Volatile Matters, Fixed Carbon, Sulfur, Calorific Value - increased as a result of dewatering and addition of petroleum residuum.

C, H, N - Increased as a result of dewatering and addition of residuum.

O - Decreased as a result of thermal decomposition and addition of residuum.

Hardgrove Grindability Index (HGI) - Only small changes in the Hardgrove Grindability Index. There were no difficulties in testing these samples using ASTM specification D409-71.

Residuum Content - An average of 9.5% for Wabamun and 15.2% for Poplar River. These again confirm that lignites require more residuum than subbituminous coals. These figures are also higher than those obtained from batch operation, because of the shorter residence time in the draining process than in the batch process.

Equilibrium Moisture and Porosity - The degrees of reduction in equilibrium moisture and porosity are consistent with those observed in Phase I (ca. 60%).

Tendency for Spontaneous Combustion - Products show lower tendency for spontaneous combustion as shown in Figure 3.

7. PROCESS ECONOMICS

The subject of process economics (Task 11R) is eliminated in this final report. This is based on the fact that data of mass and heat balances cannot be established during the short period of continuous operation, and that technical feasibility of the two continuous processing units remains questionable.

8. CONCLUSIONS AND OVERALL ASSESSMENT

Upgrading low rank coals with heavy residuum using the immersion technology presents no problem in batch operation. For coal particles of sizes 38.1 mm X 12.7 mm, moisture reductions are in the range of 70-95% at temperature 210-240°C and residence time 10-20 min. The reduction of water and the addition of heavy residuum, increase the heating values of subbituminous coal and lignite by 20% and 45% respectively. The results of this study, together with the results from the Phase I study, have established that coal particles of sizes 38.1 mm X 3.2 mm can be used as feedstock for this upgrading method.

The development of this process into a continuous operation, however, is not entirely satisfactory. The process works well for a short period of time (1 hour), but fails to produce products of consistent residuum level for an extended period. Products obtained in the initial period were superior in qualities and properties. They have lower water content (ca. 1%) and improved calorific values (15% higher for subbituminous coal and 34% higher for lignite). They also have lower porosity (ca. 60% reduction), lower equilibrium moisture (ca. 45% reduction), and lower tendency for spontaneous combustion (3°C temperature rise vs. 12°C for untreated coal under similar conditions).

The first continuous unit extensively evaluated in the experimental program is a rotary kiln reactor. Rotary kiln reactors are commercially available and can combine coal immersion and residuum draining in a single unit. Preliminary experiments with coal feedstocks indicated severe limitation in heat transfer which will be more serious in the scale-up version; and the model experiments with Paraflex oil and stone aggregates showed that this unit was working as a size classifier. The rotary kiln reactor also requires mechanical clean-up when process upset is encountered.

The second continuous unit studied in the experimental program was a stirred tank reactor followed by a vibrating screen system. The two-stage processing concept is closer to the concept of batch operation. The tests conducted on pitch characteristics showed that there were little changes in residuum properties which might affect screen performance; and pitch draining tests indicated that

vibrating screens are most likely to give products with acceptable residuum level. Integration of the two stages which showed great promise in continuous processing, however, gave satisfactory performance only for a relatively short period of time.

The results of the Phase II program show that, while the qualities and the handling characteristics of the beneficiated products are superior, the production of these upgraded coals by immersion technology is difficult. The unsatisfactory performance of commercial equipment, the rotary kiln and the stirred tank reactors, make the process unlikely to become commercial in the near future.

Apart from heat transfer problems and processing difficulties experienced in the commissioning of laboratory scale continuous units, it is apparent that the control of residuum level has an important impact on process operability. The study shows that the qualities and handling characteristics of the beneficiated coal prepared with heavy residua are established, but an alternative processing is required to deliver these products.

9. RECOMMENDATION FOR FUTURE WORK

The results of the Phase II program show that the draining step in the immersion technology has greatly complicated the operability of continuous processing. The injection method proposed for future development will eliminate this step, thus significantly enhancing the chance of success for commercial operation. The injection method is briefly described below.

The injection method consists of drying low rank coals concurrently by flue gas from a gas burner in a rotary drum mixer, and at the same time, injecting desired quantities of residuum by a spray nozzle onto preheated coal in the drying zone. Coal particles are coated and heating is continued to remove water from coal pores which are then sealed by the residuum. This method differs from traditional thermal dryer and ensures even dispersion of residuum on coal particles. It totally eliminates the draining step which was largely responsible for the unsatisfactory performance of the two continuous units evaluated in the Phase II program.

Preliminary investigation of this method at Gulf R&D shows that the products obtained are quite comparable with those produced from the immersion technology. Patent application for this process is in progress. This injection method is recommended for future development.

TABLE 1

ANALYTICAL DATA FOR WABAMUN AND POPLAR RIVER COAL SAMPLES

	Wabamun		Poplar River	
	38.1 mm X 25.4 mm	25.4 mm X 12.7 mm	38.1 mm X 25.4 mm	25.4 mm X 12.7 mm
Proximate				
Residual Moisture, %	12.4	12.7	24.4	25.0
Ash %	19.0	20.2	8.2	10.5
Volatile Matter, %	30.6	30.1	34.9	33.5
Fixed Carbon, %	38.0	37.0	32.5	31.0
Ultimate				
Residual Moisture, %	12.4	12.7	24.4	25.0
C %	50.4	48.5	46.7	43.3
H %	2.8	2.7	2.9	2.9
N %	0.6	0.6	0.5	0.5
S %	0.1	0.1	0.4	0.5
Ash %	19.0	20.2	8.2	10.6
O % (by difference)	14.7	15.2	16.9	17.2
Calorific Value cal/g	8091	7976	7559	7135

TABLE 2

OPERATING CONDITION AND YIELD OF PILOT PLANT THERMAL TREATMENT OF EDMONTON VTB

	DRB Reactor 51.8 m Coil	DRB Reactor 22.9 m Coil
Temperature, °C	420	425
Pressure, MPa	4.3	4.3
Residence Time (min)	50	43
Yields based on VTB Feed, wt%		
Gas	5.8	
IBP-190°C	3.1	
190-360°C	13.6	
360°C+	77.5	
EFV Unit (Equilibrium Flash Vaporization)		
Temperature, °C	330	
Pressure, KPa	0.27	
Feed Rate, kg/h	6	
Yields based on VTB Feed, wt%		
360-545°C	15.2	
545°C+	62.3	

TABLE 3

PROPERTIES OF PITCH FOR COAL BENEFICIATION

	Thermally Treated Edmonton VTB
C %	87.3
H %	9.9
N %	0.5
O %	0.9
S %	1.4
Calorific Value (cal/g)	10 028
Ni ppm	26
V ppm	38
Specific Gravity	1.0383
Viscosity (cSt) @ 150°C	817
@ 175°C	248
@ 200°C	82
Softening Point, °C	95
Penetration @ 25°C	1

TABLE 4

RESULTS OF COAL BENEFICIATION USING BATCH REACTOR

(WABAMUN COAL AND THERMALLY TREATED EDMONTON VTB)

Expt. #	Size (mm)	Reaction Temp. (°C)	Draining Temp. (°C)	Residence Time (min)	Draining Time (min)	% Yield	% RM	% Ash	% VM	% Residuum	S %	Heating Value (cal/g)
JM-7	38.1X25.4	215	180	15	10	91.0	2.5	36.7	-	6.7	0.3	5091*
JM-13	38.1X25.4	234	184	10	10	92.3	1.0	31.4	-	5.1	0.3	5636*
JM-3	38.1X25.4	238	193	10	10	86.9	3.5	20.1	-	6.7	0.3	5244
Raw Coal for JM-7, JM-13, and JM-3	38.1X25.4	-	-	-	-	-	12.4	19.0	30.6	-	0.2	4495 53.24** 18.4+
JM-8	25.4X12.7	214	190	15	10	99.7	3.4	18.2	37.0	-	0.3	5615
JM-14	25.4X12.7	230	185	10	10	90.5	0.3	31.8	-	3.8	0.3	5674*
JM-1	25.4X12.7	242	205	20	10	88.2	2.9	20.7	-	5.3	0.3	5191
JM-2	25.4X12.7	243	190	10	10	86.7	3.1	20.6	-	5.4	0.3	5184
Raw Coal for JM-8, JM-14, JM-1, and JM-2	25.4X12.7	-	-	-	-	-	12.7	20.3	30.1	-	0.2	4431 5416** 22.2+

* Adjusted to Ash % = 20

** Average Value

\+ Increase in Heating Value (Product vs. Feed)

TABLE 5

RESULTS OF COAL BENEFICIATION USING BATCH REACTOR

(POPLAR RIVER LIGNITE & THERMALLY TREATED EDMONTON VTB)

Expt. #	Size (mm)	Reaction Temp. (°C)	Draining Temp. (°C)	Residence Time (min)	Draining Time (min)	% Yield	% RM	% Ash	% VM	% Residuum	S %	Heating Value (cal/g)
JM-9	38.1X25.4	219	180	20	20	80.5	4.2	7.3	46.4	18.0	0.8	6119
JM-11	38.1X25.4	225	180	10	10	95.8	6.5	7.7	46.4	28.9	1.0	6168
JM-6	38.1X25.4	253	198	10	10	79.5	3.8	8.9	-	11.6	-	5980
Raw Coal	38.1X25.4	-	-	-	-	-	24.4	8.2	34.9	-	0.5	4199
for JM-9, JM-11, and JM-6												6089**
												45.0+
JM-12	25.4X12.7	220	180	10	10	95.4	3.8	11.6	45.2	21.5	0.8	5980
JM-10	25.4X12.7	220	180	20	20	89.9	3.3	10.3	45.3	19.4	0.8	6013
JM-5	25.4X12.7	236	203	10	10	77.4	4.6	12.5	-	10.5	0.7	5567
JM-4	25.4X12.7	244	196	20	10	73.7	3.7	12.5	-	10.9	0.7	5623
Raw Coal	25.4X12.7	-	-	-	-	-	25.1	10.6	33.5	-	0.4	3963
for JMj-12, JM-10, JM-5, and JM-4												5796**
												46.2+

** Average Value

+ Increase in Heating Value (Product vs. Feed)

TABLE 6

RESULTS OF PITCH CHARACTERIZATION TESTS

SERIES A - Steam purge (1.38 kg/h) of pitch (211°C)

Time, h	0	1	5	9	13
Brookfield Viscosity cSt @ 240°C	90	67	76	67	72
Softening Point, °C	120	119	124	113	126

SERIES B - Steam purge (1.38 kg/h) of pitch (242°C) containing 20 wt% 1.00 mm X 0 coal particles

Time, h	0	1	5	9	13
Brookfield Viscosity cSt @ 240°C	78	54	64	55	58
Softening Point, °C	108	110	111	122	123

TABLE 7

RESULTS OF DRAINING TESTS ON STATIONARY SCREENS

Series	Screen Type	Oil/Residuum	Temp. (°C)	Wt.% Coal	Coal Size (mm)	Wt. % Oil/Residuum on Coal After 0.8 min	1.8 min	30.0 min	35.0 min	65.0 min
A	Slot Wedge	Paraflex Oil	23	10	3.2X1.0	29.7	29.3	25.5	-	-
				20		30.1	32.0	25.1	-	-
				30		31.3	24.7	25.0	-	-
B	Round Hole	Paraflex Oil	23	20	3.2X1.0	30.5	29.4	28.5	-	-
C	Round Hole	Thermally treated Edmonton VTB	240	20	3.2X1.0	30.1	19.5	15.8	-	-
D	Slot Wedge	Thermally treated Edmonton VTB	240	18	3.2X1.0	-	-	-	13.4	18.7

TABLE 8

DATA OF DRAINING TEST ON SWECO VIBRATING SCREEN
(COAL SIZE: 12.7 mm X 9.5 mm)

Screen Size and Excess Weight	% Resid on Coal 1.5 min	2.5 min	5.0 min	15.0 min	30.0 min
24 mesh screen (479 g excess weight*)	6.9	5.2	6.2	7.3	5.6
24 mesh screen (1233 g excess weight*)	5.5	4.6	3.1	2.6	6.0
14 mesh screen (1233 g excess weight*)	6.7	4.7	6.0	6.5	7.9

*NOTE: Excess weight refers to weights added to the existing fixed weight on the shaker motor to provide increased vibration

TABLE 9

TEST RESULTS ON STIRRED TANK - DEDUCK SCREEN SYSTEMS

Test No.	Time min	Temperature, °C Top Gas Inlet	Bottom Gas Inlet	Bottom Drain	Screen Location 1*	2*	3*	4*	Exit Gas	Results Prod. kg/h	Resid %	Notes
1	0	355		78					148	11.8	9.5	
											7.6	
											7.2	
											7.1	
	30	357		148					158	12.0	6.9	
2	0	Cold Start										Resid Temp. 253
	30	333	355	192	145	158	161	182	115			
	60	333	365	224	177	181	209	112	155			Resid Temp. 239
	90	333	370	236	197	191	230	175	175			
	100	333	-	242	224	206	230	196	186	4.8	7.2	
	120	329	-	244	221	205	231	188	184			
		Screen channel clean throughout length except last 5 cm.										
3	0	331		246		209	232	172				Resid 250
	15	332		246	223	209	233	190	191	9.9	8.6	
	30	332		295								Resid 235
4	0	333		247	221	206	231	185	151			Screen lifted 3.8 cm, Resid 254
	15	332		246	225	212	229	193	156	11.1	8.0	
	30	328		244	223	212	217	193	158			Resid 229
5	0	333		244	217	206	213	180	154			Screen lifted to 11.4 cm, Resid 253
	15	333		245	221	211	212	196	157	14.0	7.2	
	30	334		242	222	214	212	196	158			Resid 226
6	0	365	388	258	228	215	219	191	162			8.9 cm top orifice, Resid 252
	15	365	376	256	227	218	217	197	165	13.2	6.4	Tightened screen
	30	363	376	250	225	218	209	201	166			Resid 223
7	0	355	376		212	196	217	176	190			45.7 cm wide screen, Resid 257
	15	355	385		230	199	213	177	188	12.0	6.4	Resid 230
8	0	356	386		238	203	215	73	187			Tightened screen, Resid 252
	15	356	385		236	205	216	175	190	12.4	6.2	Resid 237
9	0	356	385		241	206	216	170	186			Screen lifted to 20.3 cm, Resid 250
	15	352	385		249	218	218	178	191	12.3	5.1	Resid 236

* Screen Location
1 - 30.5 cm from inlet
2 - 91.4 cm from inlet
3 - 91.4 cm from exit
4 - 30.5 cm from exit

TABLE 10

EXPERIMENTAL CONDITIONS FOR CONTINUOUS OPERATION

Run # DS-24 (Wabamun)

Coal Feed:	7.5 kg/h
Particle Sizes:	25.4 mm X 6.4 mm
Temperatures, °C:	
Stirred Tank Reactor:	307
Inlet Gas	384-404
Exit Gas	275
Screen	
at slurry entrance	313
at exit	278

Run # DS-25 (Poplar River)

Coal Feed:	7.5 kg/h
Particle Sizes:	25.4 mm X 6.4 mm
Temperatures, °C:	
Stirred Tank Reactor	302
Inlet Gas	384-404
Exit Gas	277
Screen	
at slurry entrance	315
at exit	297

TABLE 11

QUALITIES OF PRODUCTS AND PARENT COALS CONTINUOUS OPERATION

	% H_2O	% Ash	% VM	% FC	% S	Calorific Value Cal/g	HGI	% Residuum	C %	H %	N %	O %	O % Dry Basis Ash Free
DS-24 - Continuous Run													
Raw Coal	10.0	12.0	37.1	40.9	0.3	5116	49	-	56.1	3.3	0.7	17.5	22.9
DS-24 (Product)	1.1	16.8	37.0	45.1	0.5	5906	48	9.5	62.4	4.4	0.8	14.0	17.1
DS-25 - Continuous Run													
Raw Coal	16.7	10.6	42.6	30.0	0.6	4361	40	-	48.4	3.3	0.5	20.5	28.2
DS-25 (Product)	1.6	16.0	41.2	41.2	0.7	5856	54	15.2	61.3	4.5	0.6	16.0	19.4

TABLE 12

PROPERTIES OF PRODUCTS AND PARENT COALS
CONTINUOUS OPERATION

	DS-24 (Wabamun)			DS-25 (Poplar River)		
	Raw Coal	Product	Reduction %	Raw Coal	Product	Reduction %
Equilibrium Moisture (%)	17.6	10.2	42	25.8	14.2	45
Porosity (cc/g)	0.1445	0.0902	60	0.1652	0.1044	58

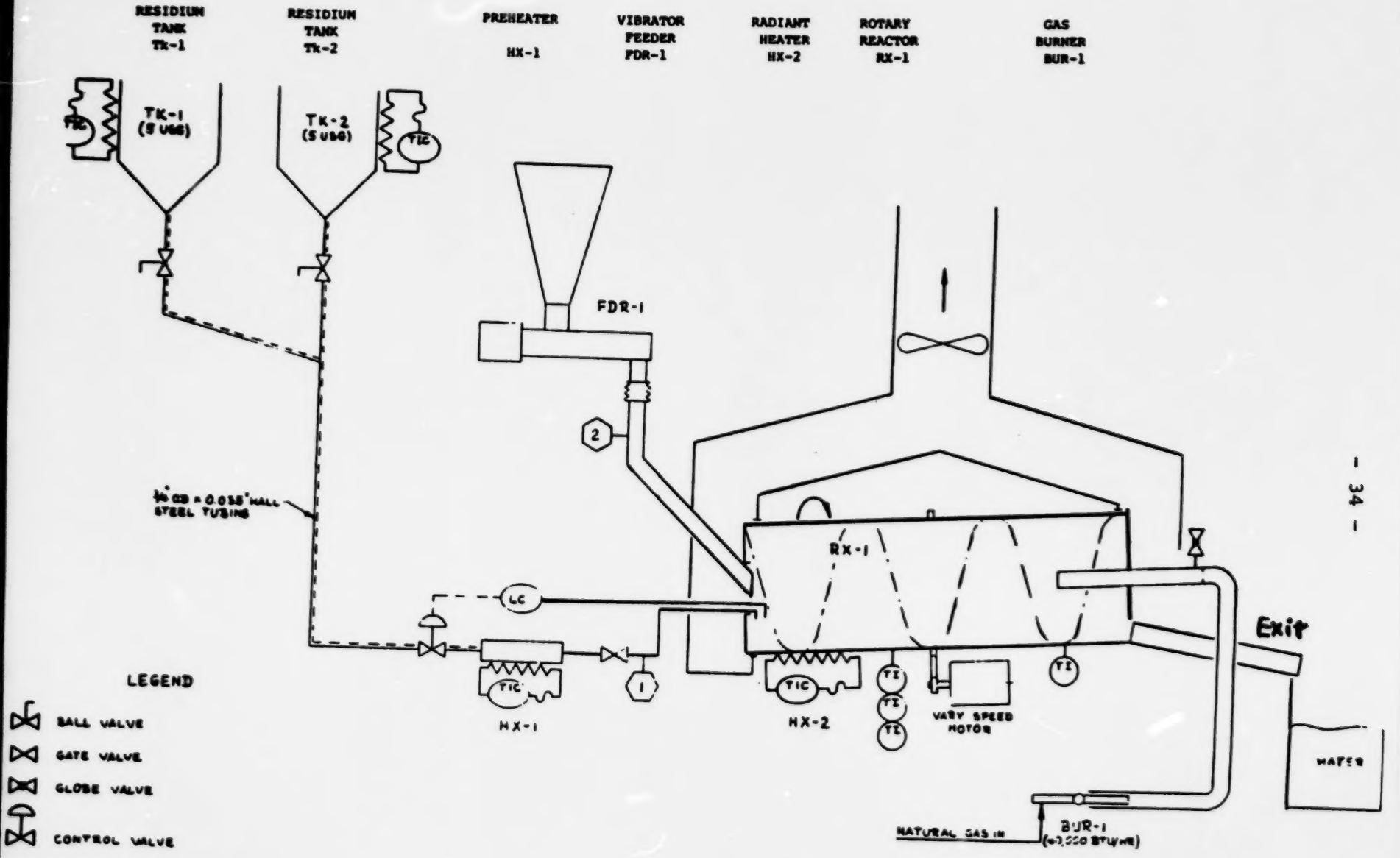


FIGURE 1 : FLOWSHEET FOR CONTINUOUS UNIT COAL BENEFICIATION

(Rotary Kiln Reactor)

FIGURE 2

FLOWSHEET FOR STIRRED TANK - VIBRATING SCREEN CONTINUOUS UNIT

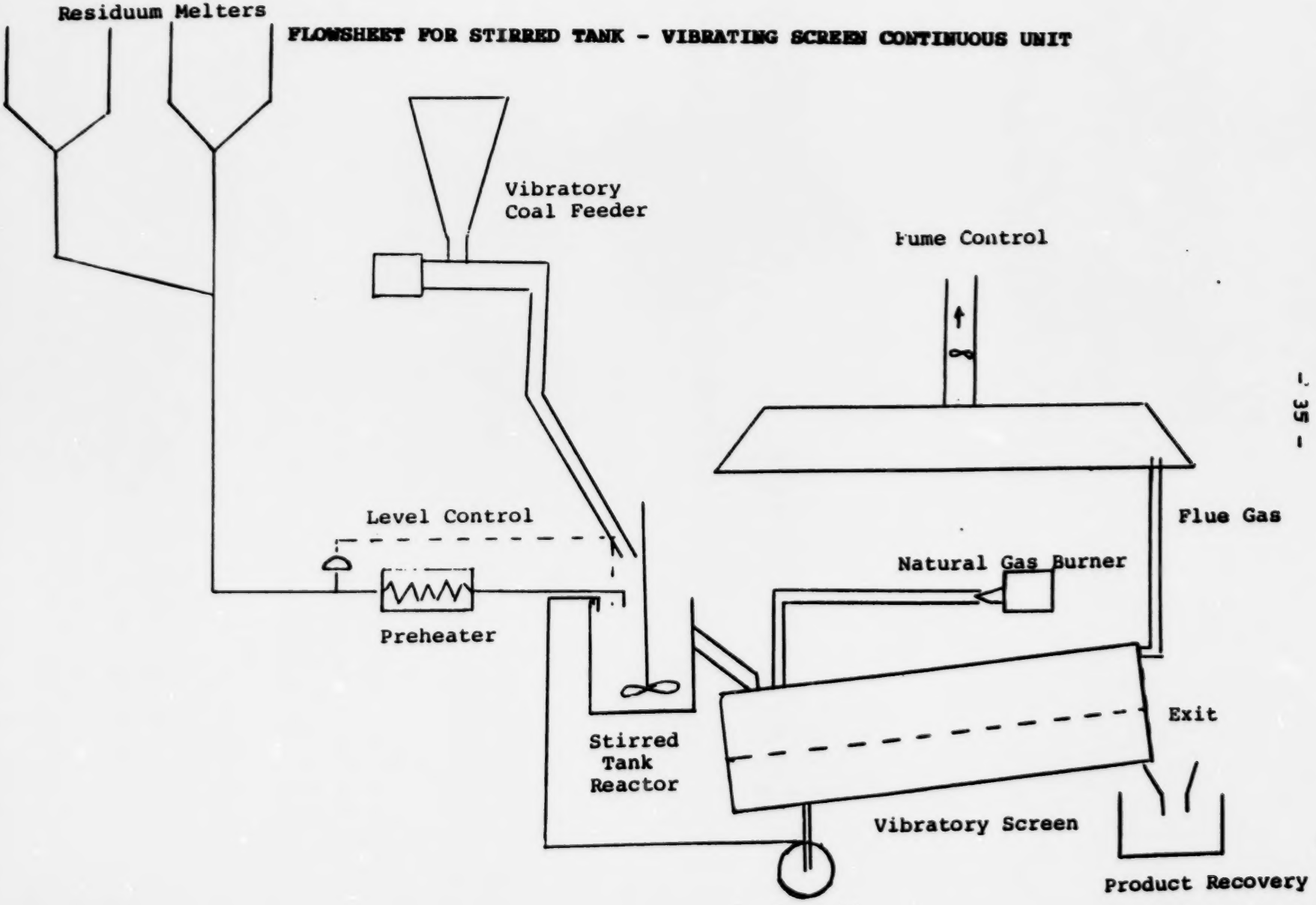
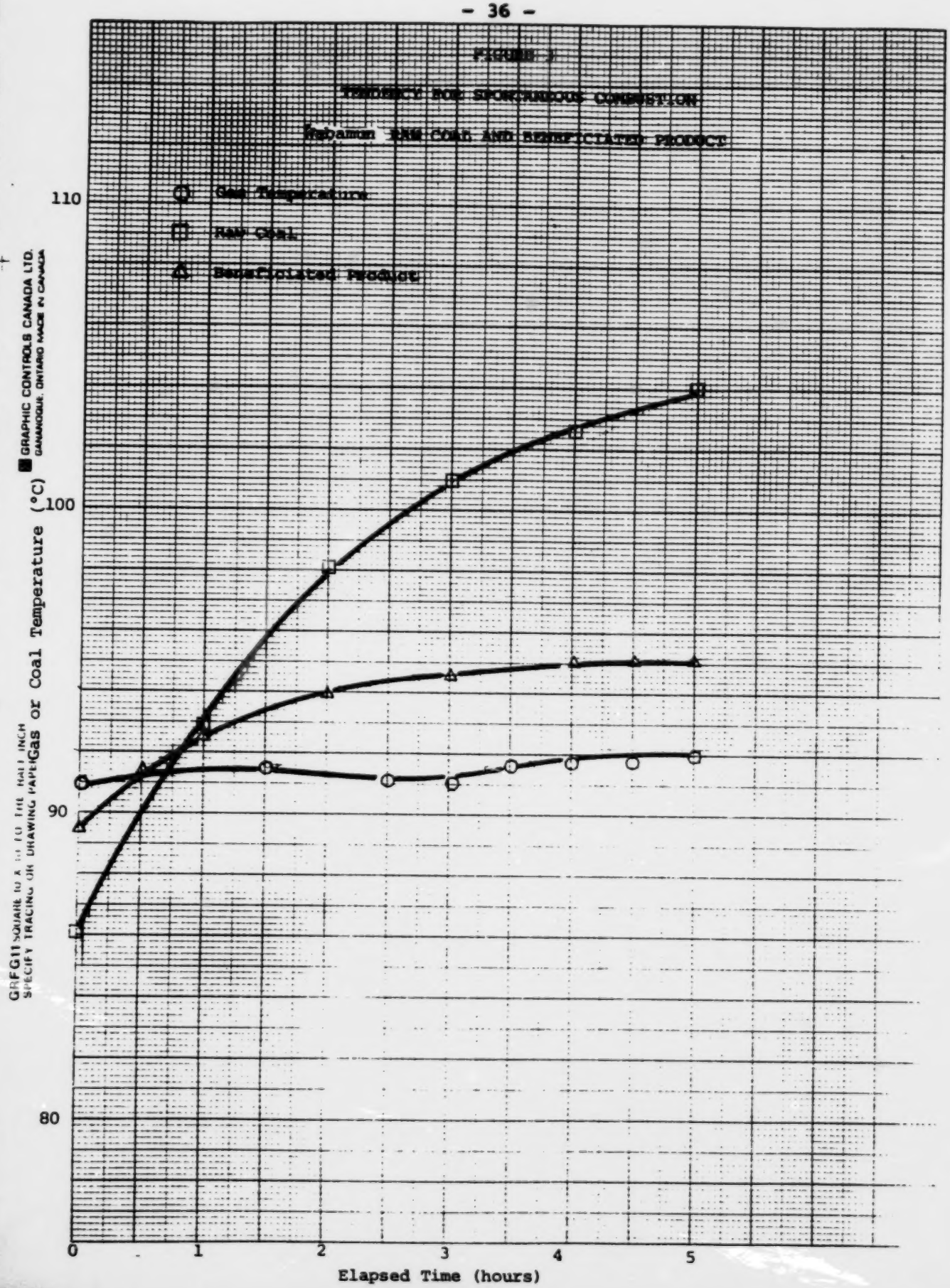


FIGURE 3

TENDENCY FOR SPONTANEOUS COMBUSTION

Kaiparowits RAW COAL AND BENEFICIATED PRODUCT



APPENDIX 1

APPENDIX 1

6. WORK STATEMENTS

Gulf Canada Limited $299.2K total value
$149.6K CANMET's share

The work that will be performed in the 2nd phase program can be divided into 13 specific tasks. This program will take 12 months to complete and the time schedule for each task is shown in the Bar Chart in Figure 1.

The coal samples recommended for the 2nd phase study are Wabumun subbituminous coal and Poplar River Lignite. These samples were acquired in the 1st phase program (9) and sufficient quantities are available. These samples have been selected because they represent important low rank coal resources available from current operations.

If the results of the Phase 1 program show that lignitic coals are less suitable for this process, another subbituminous coal will then be considered. This coal may be selected from the subbituminous coals chosen in Phase 1, or another subbituminous coal will be identified. The selection of the new feedstock will be subject to the approval by CANMET Scientific Authorities.

6.1 Task No. 1 - Acquisition of Heavy Residuum Sample

One heavy residuum sample of suitable quality will be acquired for this program. The selection will be based on the results of the first stage study and will be made by taking into account both technical and economic factors.

The quantity of appropriate quality residuum required is estimated to be about 1 to 1-1/2 barrels. If Edmonton VTB is chosen, three barrels will be required to produce sufficient quantity with suitable quality. These samples will be shipped to Gulf Canada R&D.

The selection of the residuum will be subject to approval by CANMET Scientific Authorities.

6.2 Task No. 2 - Preparation of Heavy Residua Samples

Edmonton VTB is a possible choice for the residuum to be used in the Phase 2 program. It is available from current operations and has a low sulfur content. However, it requires additional thermal treatment and fractionation to raise its softening point. The thermal treatment and fractionation will be subcontracted out to another research organization, if this residuum is chosen.

The most likely subcontractor for thermal treatment and fractionation is the Sandwell Beak Research Group in Mississauga. The thermal treatment can be conducted on a continuous unit at Sandwell.

If a residuum is chosen which requires no preparation, this task will be eliminated.

6.3 Task No. 3 - Analysis of Heavy Residua Samples

The residuum sample will have to be analyzed because this sample may be different in quality from the one analyzed in Phase 1. The following analyses will be carried out at the locations indicated.

Elemental Composition - subcontracted to Guelph Chemical Laboratories Ltd., Guelph, Ontario

Calorific value, Ni, V, API gravity, viscosity, softening point, penetration - Gulf Canada R&D

6.4 Task No. 4 - Analysis of Coal Samples

The top size of coal samples recommended for the experimental work in Phase 2 is 1 1/2". The extension (of particle size) for the Phase 1 study is important because it will expand the applicability of this upgrading process, and at the same time, reduce the cost of feedstock preparation.

The +1/2" retains of the two coal samples obtained in Phase 1 program will be screened into +1 1/2", 1 1/2" x 1", and 1" x 1/2" size fractions. Depending on the amount of the +1 1/2" material, they may be reduced to 1 1/2" top size, rescreened at the same sizings, and added to the original size fractions. These size fractions will be subjected to proximate analysis, ultimate analysis, and calorific value. This work will be sub-contracted out.

The screening of the coal samples will be sub-contracted out to either Ontario Research Foundation in Sheridan Park or Loring Laboratories Ltd. in Calgary. Coal analysis will be sub-contracted to Loring Laboratory.

This task will also cover the work to acquire a new subbituminous coal sample, if required.

6.5 Task No. 5 - Batch Experiments

The beneficiation experiments using the larger particle sizes will be run in the apparatus described in detail in the 1st phase program. Approximately 50 runs will be required. The operating conditions of the 1st phase program will be reviewed to decide the exact conditions for these experiments.

The proposed experimental conditions and the number of experiments for each coal are summarized in the following table. Any change in operating conditions required, will be subject to the approval of CANMET Scientific Authorities.

Variables	Wabumun	Poplar River
Size Fractions	2	2
Temperature of Residuum	2	2
Temperature of Draining	2	2
Residence Time	2	2
Sub-Total	16	16
Establishing Reaction Conditions & Replicates	8	8
TOTAL	24	24

6.6 Task 6 - Analysis of Products

This task consists of the analysis of products from the batch experiments (Task 5) and those from the continuous experiments (Task 9).

6.6.1 Products from Batch Experiments

The following analyses will be carried out:

50 Calorific value
50 Residuum content
50 Sulfur content
- Gulf Canada R&D

50 Proximate analysis
50 Hardgrove grindability index
25 Ultimate analysis
- Sub-contractor Laboratory

6.6.2 Products from Continuous Experiments

Fixed Interval Samples

40 Calorific value
40 Residum content
40 Sulfur content
- Gulf Canada R&D
40 Proximate analysis
- Sub-contractor Laboratory

Final Composite

10 Proximate analysis
10 Hardgrove grindability index
10 Ultimate analysis
- Sub-contractor Laboratory

Sub-contractor laboratory is Loring Laboratories Ltd. in Calgary.

6.7 Task No. 7 - Design of a Laboratory Continuous Unit

The results from Tasks No. 5 and 6 of this proposal and those from the 1st phase program will establish the optimum operating conditions, and mass and heat balances for the two selected coals. This information can be used as the basis for the design and construction of a laboratory scale continuous unit for this beneficiation process.

The capacity of this laboratory scale continuous unit will be in the order of one kg/h. Major components for this unit will be a continuous coal feeding system, a continuous residuum feeding system, a reaction chamber (immersion chamber and residuum draining chamber), a water separation unit, and a gas monitoring device. The design will also cover all necessary instrumentation for monitoring process parameters.

Careful attention will be paid to the safety aspects of this operation. The handling of hot residuum and fumes from the process merit particular attention.

6.8 Task No. 8 - Construction of a Laboratory Scale Continuous Unit

The laboratory scale continuous unit will be constructed according to an in-house design, as much as possible. Gulf Canada, therefore will be responsible for selection of equipment and material, purchasing the same, and assembly and shake-down of this unit.

6.9 Task No. 9 - Experimental Program Using Continuous Unit

The objective of this task is to examine the operability of this upgrading process in a continuous mode. Specifically, satisfactory handling of hot residuum, product, collection of water and gas from a continuous operation would add confidence to a future Pilot Plant operation.

The selected residuum and two coal samples will be run under conditions established in Phase 1 and Phase 2 programs. Five sets of experiments are proposed for each coal. These are 4 distinct size fractions (i.e., 1 1/2" x 1", 1" x 1/2", 1/2" x 3/8" and 3/8" x 1/4") and one combined size fraction (1 1/2" x 1/4"). A total of 10 sets of experiments therefore will be run in this task.

A continuous operating time from 12 to 24 hours for each set will be used. Sampling will be conducted every hour and composits of hourly samples will also be prepared. In addition, composite samples at fixed time intervals (e.g. every 6 hours) will be prepared and analyzed for product consistency. All the analyses required are tabulated in Task No. 6.

6.10 Task No. 10 - Product Evaluations

The continuous operation will generate sufficient quantities of products (10 products of about 25 lbs. or more each) for product characterization. These products will be characterized for the following parameters:

- Porosity and pore size distribution
- Capacity moisture
- Moisture uptake
- tendency for spontaneous combustion

In addition, thermogravimetric analyses (TGA) to establish the volatile matter release profile and burning profile are recommended for the products from combined size fractions and their parent coals. TGA has a potential as a method to evaluate coal combustibility. The addition of high volatile component (residuum) may help to lower the ignition temperature and the temperature of complete combustion. The TGA data would help to establish this on a preliminary basis.

6.11 Task No. 11 - Review of Economics

After the completion of Task No. 10, the preliminary cost estimates carried out in Phase 1 will be reviewed and updated. New technical data from Phase 2 will be used and any change in economic factors will be incorporated.

The scope covered in this review will be capital cost estimates, comparison of scale, determination of operating costs, production of a sensitivity analysis and an estimate of return on investment.

6.12 Task No. 12 - Preparation of Final Report

A draft of the Final Report will be prepared and submitted to the Scientific Authorities of CANMET for approval. The final report will give a detailed description of the work performed under the project title "The Beneficiation of Low Rank Canadian Coals with Heavy Residua, Phase 2" with the following information:

1. Background information of the objective, justification and usefulness of the project for Canada.

2. A detailed design for the laboratory scale continuous/ semi-continuous unit.

3. A review of economics of this beneficiation process.

4. Experimental programs as detailed in Tasks 1 to 10.

5. Results and discussion on the effect of processing variables and product qualities, and optimum conditions for each coal as supported by tables, graphs and correlations.

6. Conclusion and recommendations.

Upon approval, 10 copies of the Final Report will be submitted to the Department of Supply and Services for Documentation.

6.13 Task No. 13 - Presentation at EMR

A presentation on the results of this Research Program will be made to the Department of Energy, Mines and Resources at the completion or during the latter part of this program.

APPENDIX 2

GULF CANADA LIMITED

2489 NORTH SHERIDAN WAY, SHERIDAN PARK, ONTARIO L5K 1A8 • (416) 822-6770 • TELEX NO. 06982256

RESEARCH AND DEVELOPMENT DEPARTMENT

File No: 415-84-83 July 17, 1985

Dr. D. P. C. Fung
CANMET, Energy Research Laboratories
Department of Energy, Mines and Resources
555 Booth Street
Ottawa, Ontario
K1A 0G1

Dear Sir:

Progress Report for Contract No. OSQ84-00097
"Beneficiation of Low Rank Canadian
Coals with Heavy Residua"
DSS File No: 23SQ.23440-4-9062

Attached is our Progress Report for Contract No. OSQ84-00097 and the Contract Plan for the period of May 1 to May 31, 1985.

This status report is, as requested, a detailed review of all activities to date. Described are the successful completion of Tasks 1 to 4 dealing with the acquisition, preparation and characterization of residuum and coal samples, the construction of a 20-litre batch reactor (Task 5), and also the completion of Tasks 7 and 8 dealing with the design and construction of the continuous laboratory unit.

However, serious problems were encountered with the operation of the continuous laboratory unit (Task 9) as discussed previously with you at our meeting in Ottawa on April 15, 1985 and again during your visit to Sheridan Park on June 20, 1985. In spite of extraordinary efforts on our part, we had to concede that the single-stage approach in a rotary kiln is not suitable for the coal beneficiation process.

We have now concluded that a two-stage system is required which consists of a stirred tank for treating the coal with residuum followed by draining the excess residuum on a vibrating screen. Preliminary evaluations have shown that this approach is feasible. Upon your suggestion we have now reviewed the requirements for completion of the contract and also prepared a proposal for a revised work statement and plan. We suggest, for your considera-tion, the following action:

1. Because the single-stage rotary kiln reactor, designed and constructed for this program, showed poor residuum drainage and heat transfer characteristics it should be substituted by the more reliable two-stage system. This continuous laboratory unit consists of a stirred tank for treating the coal with residuum followed by a vibrating screen for separating the excess residuum from the coal.

2. Resolving the problems and the modifications of the continuous laboratory unit to a two-stage process have delayed the project significantly. Therefore, we ask that an extension of six months be allowed for completion of the contract.

3. In order to accommodate the completion of the key components and objective of the contract (i.e. development and operation of the continuous beneficiation process) within the remaining, allocated resources we request some minor changes of the work statements and contract plan. The proposed changes include:

   (a) reduction of the number of batch experiments from 48 to 15
   (b) the review of economics
   (c) deletion of a formal presentation at EMR

We appreciate very much your understanding, continued cooperation and support. Furthermore we will be pleased to discuss with you any suggestions or questions you may have regarding this report and the requested amendments to the contract.

Yours very truly,

GJK/gp
Attach.

G. J. Kennepohl, Project Leader
Coal Projects

cc: B. J. Meagher
A. Logan

PROGRESS REPORT

BENEFICIATION OF LOW RANK CANADIAN COALS WITH HEAVY RESIDUA PHASE 2

DSS FILE NUMBER	:	23SQ.23440-4-9062
DSS CONTRACT SERIAL NUMBER	:	OSQ84-00097
CANMET PROJECT NUMBER	:	23440-4-9062
CANMET SCIENTIFIC AUTHORITY	:	Dr. D. P. C. FUNG

GULF CANADA LIMITED
RESEARCH AND DEVELOPMENT DEPARTMENT
SHERIDAN PARK, ONTARIO

JULY 1985

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Figure 2 Photographs of Continuous Unit (Rotary Kiln)

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1. Work Statement for D.S.S. Contract OSQ84.00097

2. Assembly Drawing of MK-II Reactor

3. R. Durrant, "Operating Manual for Continuous Coal Beneficiation Unit", February 1985, Gulf R&D Internal Report.

4. Drawing TK 5321-A1

5. Drawing D-2195-RD-1

6. Drawing D-2228-RD-1

7. Drawing D-2234-RD-0

8. Drawing D-2211-RD-0

9. Drawing D-2230-RD-0

1. SUMMARY

1.1 Feedstock preparations for coal and residuum are completed. All the feedstocks are now of appropriate qualities and in sufficient quantities for this research program.

1.2 A new batch reactor (MK-II, 20 litre capacity) was designed and constructed to handle large coal particles in the batch experimental program. This reactor is now ready for use.

1.3 Design and construction of a rotary kiln reactor for the continuous beneficiation of coal in a laboratory scale was completed.

1.4 Commissioning of the rotary kiln reactor, however, gave poor drainage of residuum and the reactor was not suitable in processing coal fines. A recovery test also failed and heat transfer limitation was severe. This reactor was abandoned after a careful assessment of the problems.

1.5 Design and construction of a stirred tank reactor for continuous coal beneficiation was initiated. Basic evaluation of this concept is ongoing and results are encouraging. Vibrating screens give promising results in controlling residuum content on the product.

1.6 Actions to be taken and priorities to be given to complete this research program are recommended. A revised Work Statement is also attached and is subject to the approval of the Scientific Authority of CANMET.

2. INTRODUCTION

This Progress Report describes work performed during November 1984 to June 1985 on D.S.S. Contract No. OSQ84-00097. The primary purpose of this research program "Beneficiation of Low Rank Canadian Coals With Heavy Residua" is to develop this beneficiation method in a continuous mode of operation on a laboratory scale. The specific objectives of this contract were:

1. To prepare coal and residuum feedstocks for this research program (Tasks 1 to 4).

2. To carry out a batch experimental program for background data in processing large particles (Tasks 5 & 6).

3. To design and construct a laboratory scale continuous unit for coal beneficiation (Tasks 7 & 8).

4. To operate the laboratory scale continuous unit (Tasks 9 & 10).

5. To review the economics of coal beneficiation with heavy residua, as previously studied in D.S.S. Contract No. OSQ83-00053 (Phase I) (Task 11).

Appendix I shows the Work Statement for each Task, and the following reports discuss the progress towards achieving these objectives.

Due to unexpected difficulties experienced in commissioning the rotary kiln reactor, the progress of this research program has been severely hindered. This report, therefore, also recommends future actions to be taken in this reseach program and proposes a revised Work Statement for discussion.

3. TASKS COMPLETED

3.1 Feedstock Preparations

3.1.1 Coal Samples

Ninety-five kg of Wabumun subbituminous coal and 140 kg or Poplar River lignite were separately screened into 1 1/2" x 1" and 1" x 1/2" size fractions, by Witteck Development Inc. of Mississauga. These size fractions were subsequently analyzed by Loring Laboratories Ltd. in Calgary. The data of proximate and ultimate analyses, and heaving values of these coal samples are shown in Table 1.

3.1.2 Residuum Sample

The Vacuum Tower Bottoms (VTB) from Gulf's Edmonton Refinery was selected for this program. Since Edmonton VTB has too low a softening point (45°C) to be used for coal beneficiation, thermal treatment to generate a pitch (504°C+ or higher cut point fraction after thermal cracking) was used to increase the softening point.

Based on thermal cracking data generated from autoclave study in the D.S.S. Contract No. OSQ83-00053, the thermal treatment of 5 barrels of Edmonton VTB was carried out in Gulf's pilot plant at Sheridan Park. The DRB (Donor Refined Bitumen) unit was used for this task. The reactor effluent was distilled to 360°C in the vacuum tower in the DRB unit. The reactor temperature for thermal treatment was selected to give about 80 wt% of the 360°C+ product. The unit ran for a total period of 8 days.

The 360°C+ fraction was further distilled in an EFV (Equilibrium Flash Vaporization) unit to generate pitch. The cut point was adjusted to produce a pitch of 95°C softening point. This pitch is used as immersion medium for the program.

The operating conditions, product yields of pilot plant thermal cracking are summarized in Table 2. The properties of the pitch are presented in Table 3.

3.2 Design and Construction of a Laboratory Scale Continuous Unit

3.2.1 Process Description

In this process low rank coals are immersed in hot petroleum residua. Moisture and some labile oxygen are driven out from the coals and their calorific value therefore increased. The calorific value of the coal is further increased by surface coating and internal adsorption of the residuum. This residuum protects the coal product from moisture readsorption and therefore reduces the tendency of the coal to spontaneous combustion.

3.2.2 Process Variables

Five process variables, which affect coal beneficiation, have been studied in a batch operation in Phase I study. These variables are coal quality, residuum quality, coal particle size, temperature, and residence time.

In general, it can be said that the final coal product properties and yield will depend mainly on the properties of the coal feedstock and the coal particle size. The operating temperature will also affect the final product yield, while the residuum quality will affect the final product properties significantly. On the other hand, the residence time and residuum quality were found to influence the product yields only marginally, and the same applied to the temperature and residence time effect on the final product quality.

3.2.3 Design Basis

Typical operating conditions and range of operating conditions for this continuous unit are as follows:

	Typical	Range
Temperature	240°C	200-280°C
Residence Time	10 min	5-20 min
Particle Size	1 1/2" x 1/8"	1 1/2" x 1/8"
Residuum Consumption	6% wt of Coal Feed	3-10% wt
Product Yield, % wt	90	85-95
Water Recovered, % wt*	8	6-10
Gas Yield, % wt.	1/2	1/2-1

* Does not include surface water.

3.2.4 Design Philosophy

The design philosophy of the laboratory scale continuous unit is to use equipment that is proven at high capacities. This will allow easy scale-up in the future. Of the many options examined for continuous coal beneficiation, rotary kiln and stirred tank reactor are considered more favourable than other devices such as auger, drum mixer, drag classifier, bucket wheel, etc. The rotary kiln, in particular, was selected because of the possibility of immersion and draining in one single step. The stirred tank reactor followed by solid-liquid separation was to be used as a back-up system.

A flowsheet showing the laboratory scale continuous unit based on rotary kiln is presented in Figure 1. This flowsheet was used to develop detailed design and the construction of the laboratory scale continuous unit.

3.2.5 Detailed Design and Construction

The laboratory continuous unit consists of the following components:

- coal feeder
- residuum feed
- level control
- rotary kiln reactor
- temperature measurement
- gas burner
- ventilation system
- operations console

Detailed discussions of these components and an operating manual for this continuous unit are presented in an internal report "Operating Manual for Continuous Coal Beneficiation Unit" which is attached in Appendix 3.

Detailed drawings used to construct this continuous unit are summarized below and attached in Appendices 4 to 9, inclusive.

Drawing	Number	Appendix No.
Rotary Reactor Arrangement & Detail	TK5321-A1	4
Rotary Reactor Assembly	D-2195-RD-1	5
Equipment Layout	D-2228-RD-1	6
Detail of Burner Piping	D-2234-RD-0	7
Fume Control System	D-2211-RD-0	8
Exhaust Duct Details	D-2230-RD-0	9

4. TASKS IN PROGRESS

4.1 Batch Experimental Program

The Batch Experimental Program was to provide background information for larger particles which were not studied in the Phase I program. However, it became quite clear that it is necessary to construct a new reactor to carry out this program, since sampling errors were significant as coal particle sizes increase.

A larger reaction vessel (20 litre capacity, Mark II) was therefore designed and constructed for this task. This reaction vessel is basically similar to the one used in the Phase 1 study but with additional stirring mechanism to ensure uniform temperature profile of the residuum. The construction of this reaction vessel was delayed by the construction of the laboratory scale continuous unit. Consequently, the batch experimental program was not carried out in this period.

An assembly drawing of the MK II reactor is attached in Appendix 2.

4.2 Operation of Laboratory Scale Continuous Unit

Commissioning tests of the rotary kiln reactor using various coals and stone aggregates indicate this reactor gives poor drainage and cannot be used to process coal fines. A recovery test and limitations in heat transfer also indicate that work on the rotary kiln reactor should be stopped.

Preliminary test runs in the continuous laboratory unit with the kiln rotating at 0.3 rpm, which gives a combined soaking and draining time of 25 minutes in the kiln showed that:

(a) that residuum was conveyed to the top of the kiln and produced together with the coal product.

(b) that finer coal fractions (e.g. 1/4" x 1/8") were retained in the kiln which acted as a size classifier.

(c) that coal was stuck with the pitch to the kiln walls and flights and could not be released upon extended heating.

(d) that the coal feed rate had to be reduced to 2.5 kg/h, only 10-15% of the design coal feed rate, in order to maintain the nominal kiln temperature of 200°C.

The observed difficulties led to the modification and redesign of the continuous laboratory unit. It became obvious that the coal treatment (soaking in residuum) had to be carried out separately from the draining of excess residuum.

5. PROPOSAL FOR REVISED WORK STATEMENT

The failure of the rotary kiln reactor designed and built for this program has significantly delayed the progress of this research program. In order to accomplish the main objective of this program, scope of the work has to be redefined and priority reset for the future work.

The data collected so far in this project suggest that the evaluation of the stirred tank reactor and the vibrating screen should be continued and be given top priority. The batch experimental program, which was not investigated for large coal particles, should also be carried out but on a reduced basis.

Review of economics can probably be deleted to give more attention to the other activities, without great sacrifice of the project objectives.

A revised Work Statement and Cost are proposed below and an extension of 6 months is required to complete this research program. The letter R is used to denote a revision and the revised tasks will replace the original tasks in the research contract.

Tasks 5R and 6R ($25,000) - Batch Experimental Program

The MK-II reactor will be used to study the effect of coal particle size on beneficiation of Wabumun and Poplar River coals. Instead of the scheduled 48 experiments only approximately 20 runs are anticipated. The commissioning of this new reactor will also be included in these tasks.

Products from beneficiation will be subjected to proximate analysis, residuum content, sulfur content, and calorific value determinations. Certain selected samples will also be subjected to ultimate analysis and Hardgrove Grindability Index determination.

Attempts will also be made to investigate the possibility of using this umit to generate data of design importance such as heat requirement, coal fines production, and changes of residuum qualities.

Tasks 7R and 8R (120,000) - Design and Construction of Continuous Unit

The revised cost for the construction of the continuous laboratory unit is $120,000. This task has been completed.

Tasks 9R and 10R ($60,000) - Operation of Continuous Unit

Evaluation of the stirred tank reactor and vibrating screen separation will be continued with the objective to process both the Wabumun and Poplar River coal in a continuous operation. The following activities are recommended:

- A study of the operation of stirred tank reactor (heat transfer and agitation required) and changes in properties of coal and residuum.
- Performance evaluation of vibrating screens in controlling final residuum content of the product.
- Laboratory scale continuous operation using Wabumun and Poplar River coals (one combined size fractions for each sample) to generate approximately 25 kg sample each.
- Product evaluations for proximate, ultimate analyses, heating value, residuum content determinations, equilibrium moisture, porosity and tendency for spontaneous combustion.

Task 11R ($5,000) - Review of Economics

The scope covered in this review will be capital cost estimates, determination of operating costs, and an estimate or return on investment.

Task 12R ($5,000) - Preparation of Final Report

A final report will be prepared containing all the data and interpretation of data.

Task 13R - Presentation

Deleted.

TABLE 1

ANALYTICAL DATA FOR WABUMUN AND POPLAR RIVER COAL SAMPLES

	Wabumun		Poplar River	
	1 1/2"x1"	1"x1/2"	1 1/2"x1"	1"x1/2"
Proximate				
RM %	12.41	12.68	24.43	25.05
Ash %	19.01	20.23	8.17	10.55
VM %	30.61	30.07	34.85	33.48
FC %	37.97	37.02	32.55	30.92
Ultimate				
RM %	12.41	12.68	24.43	25.05
C %	50.40	48.44	46.68	43.28
H %	2.80	2.71	2.94	2.88
N %	0.65	0.63	0.45	0.50
S %	0.12	0.12	0.41	0.47
Ash %	19.01	20.23	8.17	10.55
O % (by difference)	14.61	15.19	16.92	17.27
Calorific Value cal/g	8091	7976	7559	7135

TABLE 2

OPERATING CONDITIONS AND YIELD STRUCTURES OF PILOT PLANT THERMAL TREATMENT OF EDMONTON VTB

	170' Coil	75' Coil
Temperature, °C	420	425
Pressure, psig	600	600
Residence Time (min)	50	43
Yields based on VTB Feed wt%		
Gas	5.8	
IBP-190°C	3.1	
190-360°C	13.6	
360°C+	77.5	
EFV Unit		
Temperature, °C	330	
Pressure, mm Hg	2.0	
Feed rate, kg/h	6	
Yields based on VTB Feed wt%		
360-545°C	15.2	
545°C+	62.3	

TABLE 3

PROPERTIES OF PITCH FOR COAL BENEFICIATION

	Thermally Treated Edm. VTB
C, %	87.3
H, %	9.9
N, %	0.5
O, %	0.9
S, %	1.4
CV (BTU/lb;)	18 052
Ni, ppm	26
V, ppm	38
Specific Gravity	1.0383
Viscosity (cSt) @ 150°C	817
@ 175°C	248
@ 200°C	82
Softening Point, °C	95
Penetration @ 25°C	1

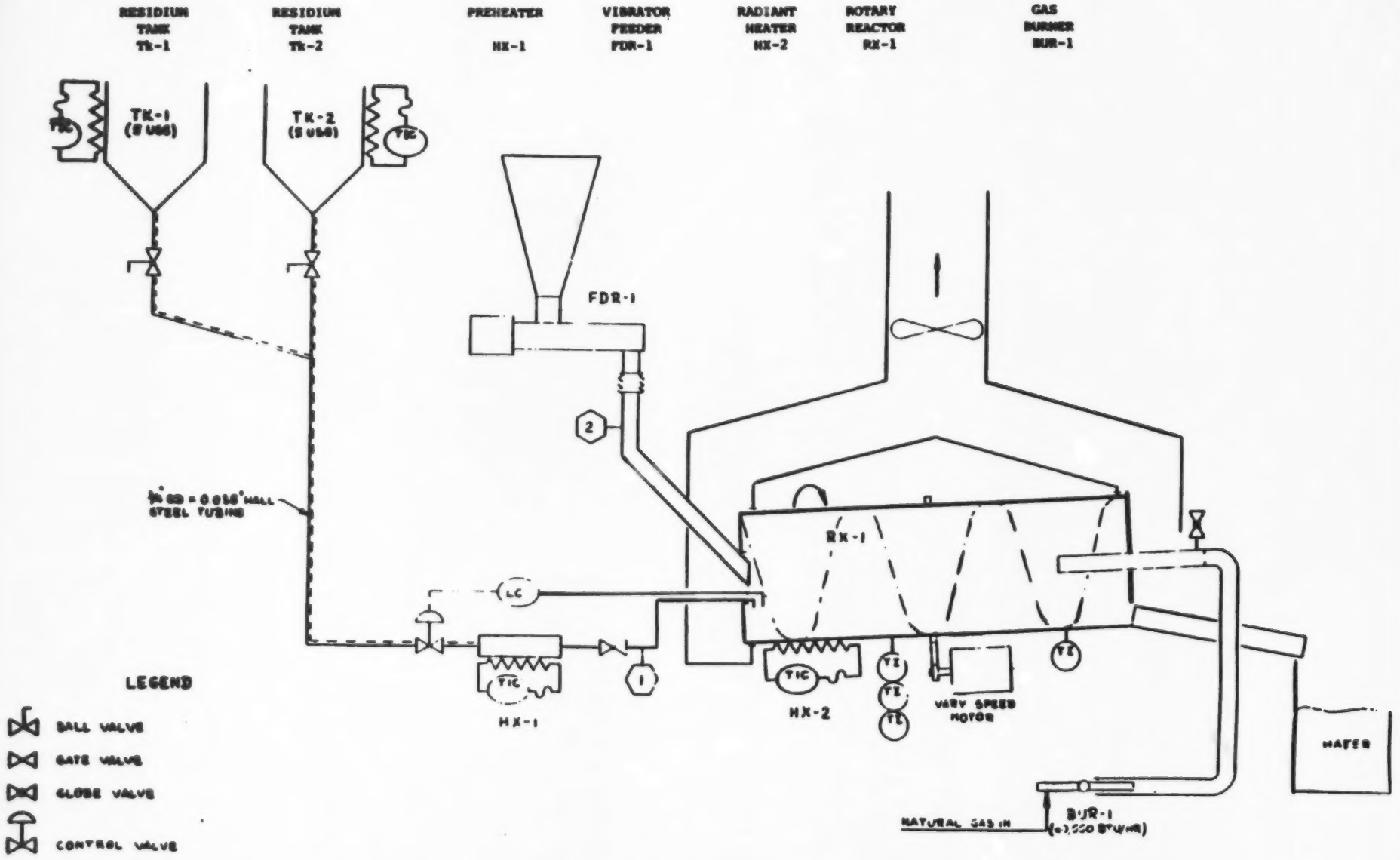
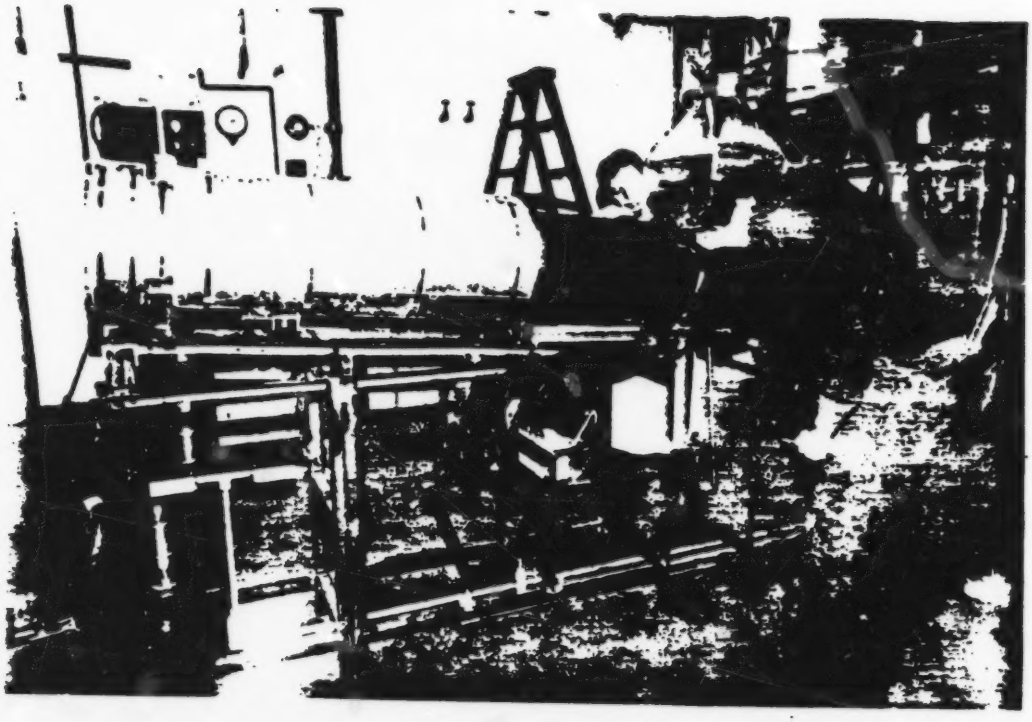


FIGURE 1 : FLOWSHEET FOR CONTINUOUS UNIT COAL BENEFICIATION



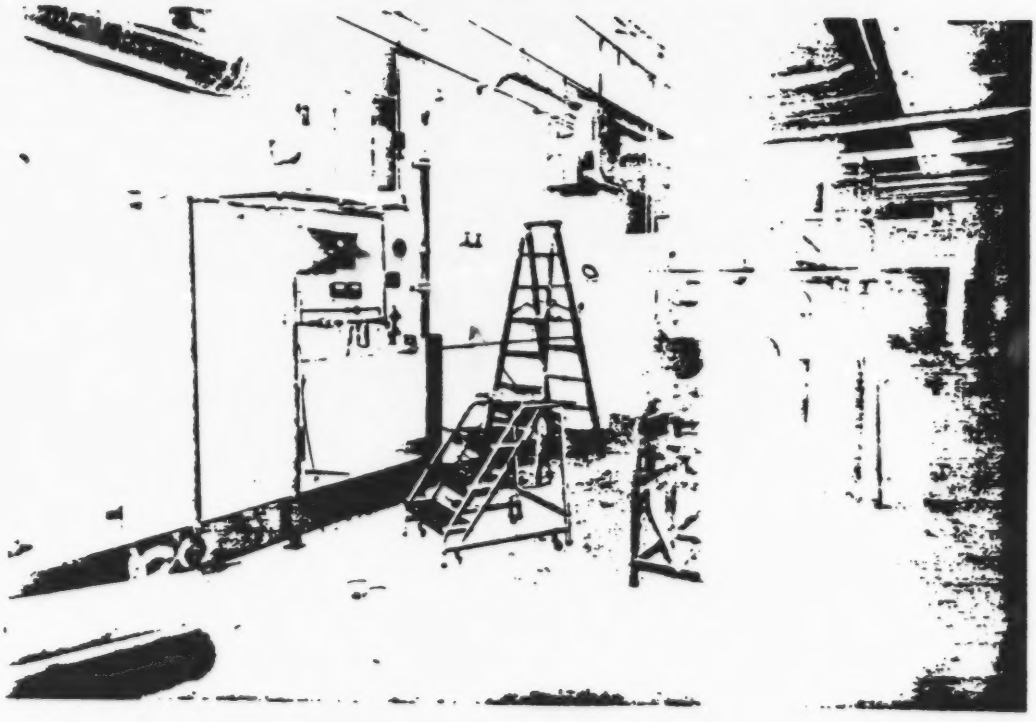


Figure 2(a): Photographs of Continuous Laboratory Unit

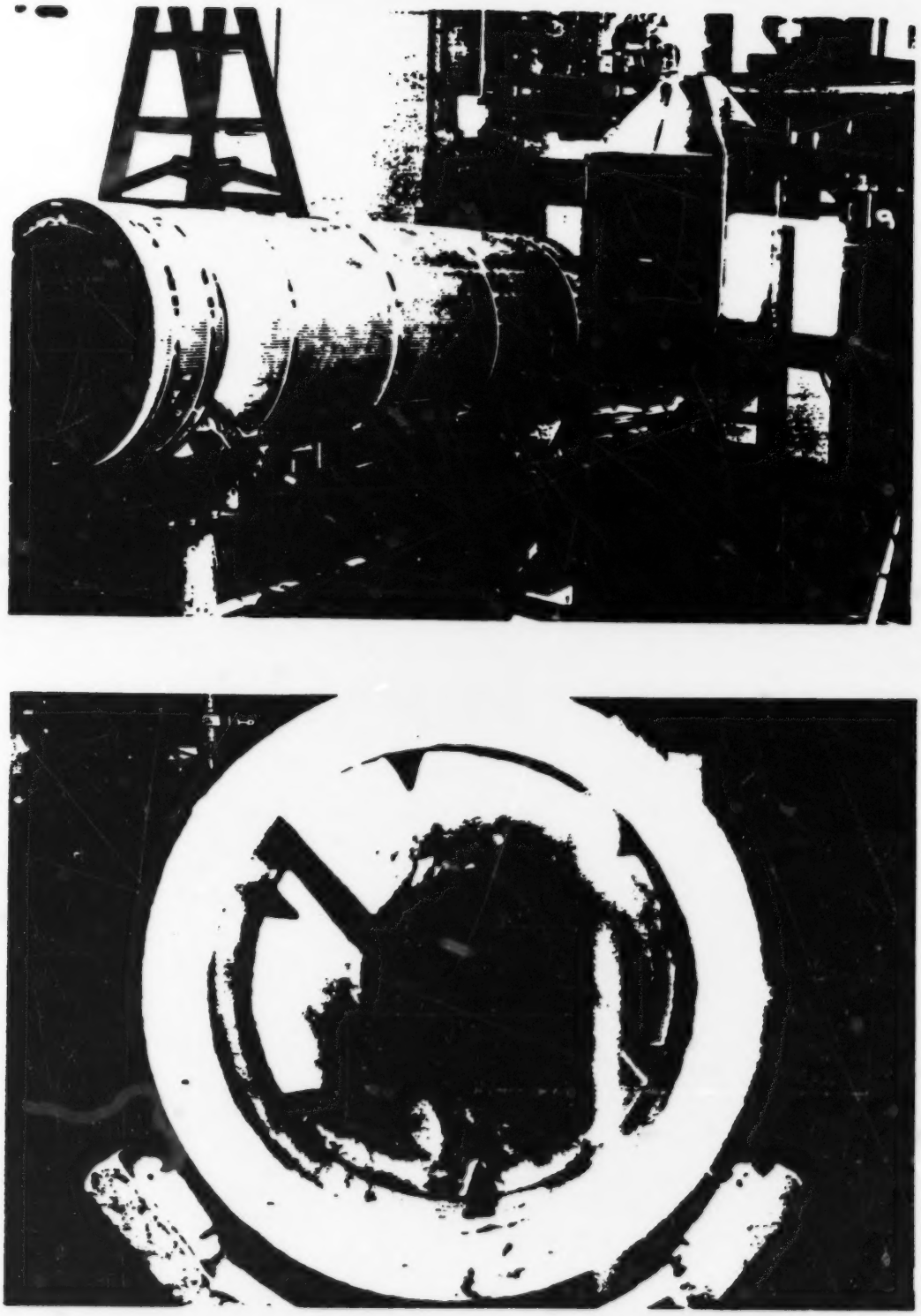


Figure 2(b): Photographs of Continuous Laboratory Unit

Figure 4(c): Photographs of Continuous Laboratory Unit

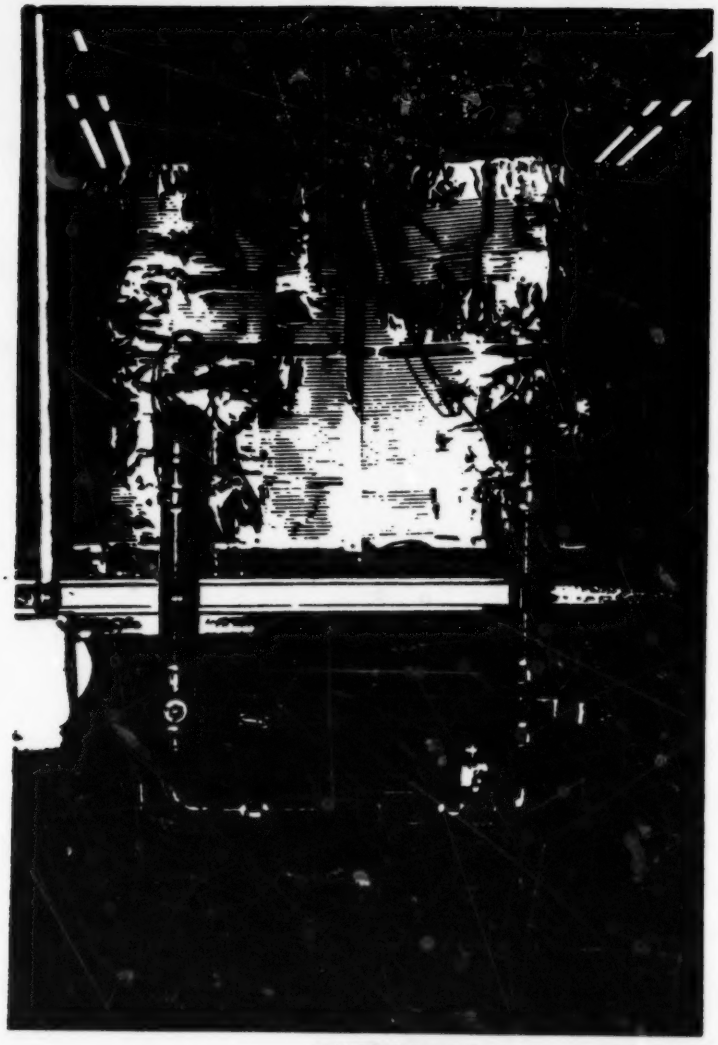
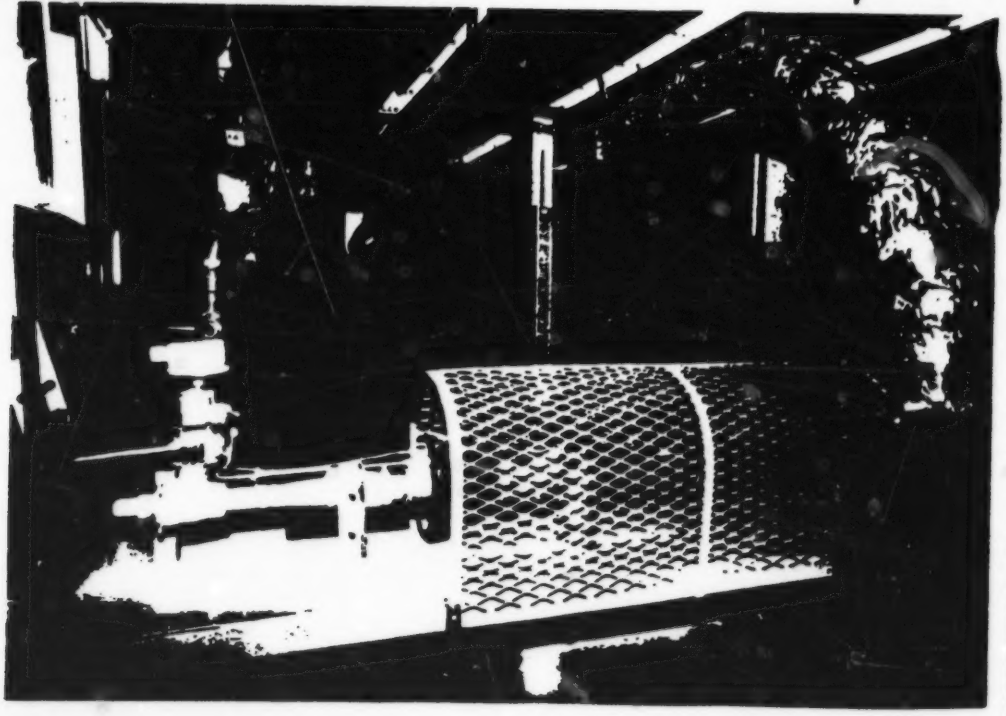


Figure 2(d): Photographs of Continuous Laboratory Unit



APPENDIX 1

WORK STATEMENTS

Gulf Canada Limited $299.2K total value
$149.6K CANMET's share

The work that will be performed in the 2nd phase program can be divided into 13 specific tasks. This program will take 12 months to complete and the time schedule for each task is shown in the Bar Chart in Figure 1.

The coal samples recommended for the 2nd phase study are Wabumun subbituminous coal and Poplar River Lignite. These samples were acquired in the 1st phase program (9) and sufficient quantities are available. These samples have been selected because they represent important low rank coal resources available from current operations.

If the results of the Phase 1 program show that lignitic coals are less suitable for this process, another subbituminous coal will then be considered. This coal may be selected from the subbituminous coals chosen in Phase 1, or another subbituminous coal will be identified. The selection of the new feedstock will be subject to the approval by CANMET Scientific Authorities.

1 Task No. 1 - Acquisition of Heavy Residuum Sample

One heavy residuum sample of suitable quality will be acquired for this program. The selection will be based on the results of the first stage study and will be made by taking into account both technical and economic factors.

The quantity of appropriate quality residuum required is estimated to be about 1 to 1-1/2 barrels. If Edmonton VTB is chosen, three barrels will be required to produce sufficient quantity with suitable quality. These samples will be shipped to Gulf Canada R&D.

The selection of the residuum will be subject to approval by CANMET Scientific Authorities.

2 Task No. 2 - Preparation of Heavy Residua Samples

Edmonton VTB is a possible choice for the residuum to be used in the Phase 2 program. It is available from current operations and has a low sulfur content. However, it requires additional thermal treatment and fractionation to raise its softening point. The thermal treatment and fractionation will be subcontracted out to another research organization, if this residuum is chosen.

The most likely subcontractor for thermal treatment and fractionation is the Sandwell Beak Research Group in Mississauga. The thermal treatment can be conducted on a continuous unit at Sandwell.

If a residuum is chosen which requires no preparation, this task will be eliminated.

.3 Task No. 3 - Analysis of Heavy Residua Samples

The residuum sample will have to be analyzed because this sample may be different in quality from the one analyzed in Phase 1. The following analyses will be carried out at the locations indicated.

Elemental Composition - subcontracted to Guelph Chemical Laboratories Ltd., Guelph, Ontario

Calorific value, Ni, V, API gravity, viscosity, softening point, penetration - Gulf Canada R&D

4 Task No. 4 - Analysis of Coal Samples

The top size of coal samples recommended for the experimental work in Phase 2 is 1 1/2". The extension (of particle size) for the Phase 1 study is important because it will expand the applicability of this upgrading process, and at the same time, reduce the cost of feedstock preparation.

The +1/2" retains of the two coal samples obtained in Phase 1 program will be screened into +1 1/2", 1 1/2" x 1", and 1" x 1/2" size fractions. Depending on the amount of the +1 1/2" material, they may be reduced to 1 1/2" top size, rescreened at the same sizings, and added to the original size fractions. These size fractions will be subjected to proximate analysis, ultimate analysis, and calorific value. This work will be sub-contracted out.

The screening of the coal samples will be sub-contracted out to either Ontario Research Foundation in Sheridan Park or Loring Laboratories Ltd. in Calgary. Coal analysis will be sub-contracted to Loring Laboratory.

This task will also cover the work to acquire a new subbituminous coal sample, if required.

5 Task No. 5 - Batch Experiments

The beneficiation experiments using the larger particle sizes will be run in the apparatus described in detail in the 1st phase program. Approximately 50 runs will be required. The operating conditions of the 1st phase program will be reviewed to decide the exact conditions for these experiments.

The proposed experimental conditions and the number of experiments for each coal are summarized in the following table. Any change in operating conditions required, will be subject to the approval of CANMET Scientific Authorities.

Variables	Wabumun	Poplar River
Size Fractions	2	2
Temperature of Residuum	2	2
Temperature of Draining	2	2
Residence Time	2	2
Sub-Total	16	16
Establishing Reaction Conditions & Replicates	8	8
TOTAL	24	24

6 Task 6 - Analysis of Products

This task consists of the analysis of products from the batch experiments (Task 5) and those from the continuous experiments (Task 9).

6.6.1 Products from Batch Experiments

The following analyses will be carried out:

50 Calorific value
50 Residuum content
50 Sulfur content
- Gulf Canada R&D

50 Proximate analysis
50 Hardgrove grindability index
25 Ultimate analysis
- Sub-contractor Laboratory

6.6.2 Products from Continuous Experiments

Fixed Interval Samples

40 Calorific value
40 Residum content
40 Sulfur content
- Gulf Canada R&D

40 Proximate analysis
- Sub-contractor Laboratory

Final Composite

10 Proximate analysis
10 Hardgrove grindability index
10 Ultimate analysis
- Sub-contractor Laboratory

Sub-contractor laboratory is Loring Laboratories Ltd. in Calgary.

7 Task No. 7 - Design of a Laboratory Continuous Unit

The results from Tasks No. 5 and 6 of this proposal and those from the 1st phase program will establish the optimum operating conditions, and mass and heat balances for the two selected coals. This information can be used as the basis for the design and construction of a laboratory scale continuous unit for this beneficiation process.

The capacity of this laboratory scale continuous unit will be in the order of one kg/h. Major components for this unit will be a continuous coal feeding system, a continuous residuum feeding system, a reaction chamber (immersion chamber and residuum draining chamber), a water separation unit, and a gas monitoring device. The design will also cover all necessary instrumentation for monitoring process parameters.

Careful attention will be paid to the safety aspects of this operation. The handling of hot residuum and fumes from the process merit particular attention.

8 Task No. 8 - Construction of a Laboratory Scale Continuous Unit

The laboratory scale continuous unit will be constructed according to an in-house design, as much as possible. Gulf Canada, therefore will be responsible for selection of equipment and material, purchasing the same, and assembly and shake-down of this unit.

9 Task No. 9 - Experimental Program Using Continuous Unit

The objective of this task is to examine the operability of this upgrading process in a continuous mode. Specifically, satisfactory handling of hot residuum, product, collection of water and gas from a continuous operation would add confidence to a future Pilot Plant operation.

The selected residuum and two coal samples will be run under conditions established in Phase 1 and Phase 2 programs. Five sets of experiments are proposed for each coal. These are 4 distinct size fractions (i.e., 1 1/2" x 1", 1" x 1/2", 1/2" x 3/8" and 3/8" x 1/4") and one combined size fraction (1 1/2" x 1/4"). A total of 10 sets of experiments therefore will be run in this task.

A continuous operating time from 12 to 24 hours for each set will be used. Sampling will be conducted every hour and composite of hourly samples will also be prepared. In addition, composite samples at fixed time intervals (e.g. every 6 hours) will be prepared and analyzed for product consistency. All the analyses required are tabulated in Task No. 6.

10 Task No. 10 - Product Evaluations

The continuous operation will generate sufficient quantities of products (10 products of about 25 lbs. or more each) for product characterization. These products will be characterized for the following parameters:

- Porosity and pore size distribution
- Capacity moisture
- Moisture uptake
- tendency for spontaneous combustion

In addition, thermogravimetric analyses (TGA) to establish the volatile matter release profile and burning profile are recommended for the products from combined size fractions and their parent coals. TGA has a potential as a method to evaluate coal combustibility. The addition of high volatile component (residuum) may help to lower the ignition temperature and the temperature of complete combustion. The TGA data would help to establish this on a preliminary basis.

11 Task No. 11 - Review of Economics

After the completion of Task No. 10, the preliminary cost estimates carried out in Phase 1 will be reviewed and updated. New technical data from Phase 2 will be used and any change in economic factors will be incorporated.

The scope covered in this review will be capital cost estimates, comparison of scale, determination of operating costs, production of a sensitivity analysis and an estimate of return on investment.

12 Task No. 12 - Preparation of Final Report

A draft of the Final Report will be prepared and submitted to the Scientific Authorities of CANMET for approval. The final report will give a detailed description of the work performed under the project title "The Beneficiation of Low Rank Canadian Coals with Heavy Residua, Phase 2" with the following information:

1. Background information of the objective, justification and usefulness of the project for Canada.

2. A detailed design for the laboratory scale continuous/ semi-continuous unit.

3. A review of economics of this beneficiation process.

4. Experimental programs as detailed in Tasks 1 to 10.

5. Results and discussion on the effect of processing variables and product qualities, and optimum conditions for each coal as supported by tables, graphs and correlations.

6. Conclusion and recommendations.

Upon approval, 10 copies of the Final Report will be submitted to the Department of Supply and Services for Documentation.

13 Task No. 13 - Presentation at EMR

A presentation on the results of this Research Program will be made to the Department of Energy, Mines and Resources at the completion or during the latter part of this program.

APPENDIX 2

ASSEMBLY DRAWING OF MK II REACTOR

APPENDIX 3

OPERATING MANUAL FOR CONTINUOUS COAL BENEFICIATION UNIT

FEBRUARY, 1985

By

R. Durrant

GULF CANADA LIMITED
RESEARCH AND DEVELOPMENT DEPARTMENT
SHERIDAN PARK, ONTARIO

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I Process Description

This process is a method of feeding coal into a rotating hellecoid conveyor, that is partly filled with hot residium. (See Figure 1) The hot residium drives the moisture out of the caol and the residium coats and is absorbed by the coal. The coal is conveyed along the rotary drum which is tilted at an angle of 5° to 15° from horizontal. The coal is exposed to the hot residium for approximately one half of the total drum length and exposed to hot gas for the remaining drum length where drainage of excess residium occurs.

The coal emerges from the rotating conveyor and is immediately cooled. The coal has been processed to prevent any future moisture absorption.

II Pilot Plant Unit Components

This operating manual describes the apparatus used to carry out on a Pilot Plant scale the process described above.

A description of the various pieces of apparatus which make up the unit follows:

a) Coal Feeder

A vibrating type Coal Feeder is used to introduce 3/8" to 1" particle size coal at a uniform rate from a 3 cubic feet hopper into a 2 7/8"^{D} coal chute set at an angle of 45°. The feeder has a variable feed rate of 2.7 CF/H Max. which is adjusted by a control located on the side of the feeder support.

b) Residium Feed

Residium is placed into two elevated vessels where the residium is melted and heated to a temperature of 150°C. One vessel is used for melting while the remaining vessel is used to feed the process with switching of feeds carried out with a pair of ball valves. The vessel temperature is controlled with a temperature controller mounted on the operator console. An over-temperature cut-out is located on the side of each feed vessel set at 180°C. The cut-out has to be reset manually by means of a

button located on the thermal cutout whenever the set temperature has been exceeded.

The feed lines from the vessels are heat traced and temperature controlled.

c) Level Control

The level of residium inside the rotating conveyor is controlled by means of a bubbler tube. (See Figure 2) Nitrogen is supplied from a nitrogen cylinder through a standard nitrogen regulator.

NOTE: the output from this regulator must not exceed 200 PSIG

The reason for this is that the regulator which follows has a maximum inlet pressure rating of 250 PSIG. A pressure of 20 PSIG is all that is required from the high pressure regulator. The low pressure regulator is set at 20 inches of water pressure. A bubbler indicator is provided to indicate rate of flow of nitrogen.

A differential pressure switch is used to sense the pressure change inside the level dip tube which varies as the residium level changes. The switch is set at 0.3" H_2O. Pressures below this setpoint cause the switch to trip which in turn causes an air operated ball valve to activiate and feed residium into the rotating conveyor. The low side of the differential pressure switch senses and compensates for changes in the pressure above the residium.

The time lag required to build pressure prevents response to turbulance and frothing inside the rotating conveyor.

Should the dip tube become plugged a second bubbler indicator set at approximately 1.5" H_2O will start to bubble and vent the nitrogen to atmosphere.

d) Rotary Reactor Conveyor

The rotary reactor consists of a 12"^{D} pipe that is 6 feet long. A helix with the center 4"^{D} removed and a pitch of 6 5/32"

long is welded at its periphery to the inside of the pipe (See FP-1527-RD for details). The rotary reactor is inclined at about 6.5° from horizontal.

The reactor is rotated at speeds of 0.075 to 0.75 r.p.m. by means of a variable speed D.C. motor and gear reducer with a chain drive.

e) Temperature Measurement

Thermocouples are placed at approximately 6" intervals along the length of the reactor (See drawing D-2224-RD-1 for details). The thermocouples protuide about 1/2" inside the reactor and are type K. The indicator and thermocouple switch rotate with the reactor eleminating the need for exotic alternatives. Reading the temperature as the indicator rotates is made possible due to the very low rotary speed of the reactor. The indicator has its own integral battery which will last for 2000 hours of continuous operation.

f) Gas Burner

The heat to maintain the rotary conveyor at process temperature is generated by a gas burner. The burner has an orifice set at 60 000 BTU. The hot gases from combustion are ducted by means of a flexible duct into the center $4"^{D}$ cavity in the rotary reactor. The duct is moveable so that the discharge point of the hot gas can be relocated inside the rotary conveyor.

The burner is fitted with a standard pilot light and automatic thermocouple shutoff should the pilot light be extinguished.

A pressure switch is included in the gas line to sense insufficient gas pressure and cut off the flow of gas to the burner by means of a solenoid valve, in the gas supply line.

g) Ventilation System

There are a total of three ventilation fans used with this apparatus.

The first is a 3/4 H.P. fan which draws the hot combustion gases

through the rotary conveyor and exhausts these gases up a stack to atmosphere. This fan is dedicated to this purpose to eliminate variations in draft setting that would occur if the fan had to serve multiple tasks.

The second fan is a 5 H.P. unit which draws from two fumehoods and a number of elephant trunks which are located as needed at the coal shaker, residium vessels and rotary conveyor outlet.

The third fan is an air make-up fan which draws air in from outside into the Coal Laboratory through a steam heat-exchanger coil. The steam valves are automatically controlled to operate when cold outside air is being introduced to the lab.

All three fans are interlocked and must be on before the main power to the Continuous Coal Unit can be switched on.

The large quantity of air moved by the fans ensures that no build-up of hazardous gases can occur.

h) Combustible Gas Detectors

A combustible gas detector with three sense points has been included as an additional safety measure. One sensor is used to monitor continuously, a sample stream from the exhaust fan which draws combustion gases out of the inside of the rotary conveyor.

A second sensor is used to monitor air withdrawn from a duct which encloses a glass solvent drain line which passes through the coal laboratory.

A third sensor is attached to a 25 foot cord that can be located anywhere in the vicinity of the Continuous Coal Unit and used as required.

The monitor is set to alarm at 20% of the lower explosive limit for methane.

i) Radiant Heaters

A set of three radiant heaters has been placed under the feed end of the rotary conveyor to provide additional heat to the pool of residium inside the rotary conveyor.

j) Operators Console

An operators console has been provided to accomodate the start-stop switches, indicator lights, temperature indicator and switch and temperature controllers. The bottom section of the console houses the solid-state relays, timers and contactors.

Note: The bottom panel door should be kept locked and only opened by electricians carrying out repairs. There are no operating controls located inside and the operator should have no need to gain entry.

The various switches are labeled with their function.

III Start-Up Procedure

The following start-up procedure is recommended in order to begin operation in a safe and logical way.

a) Turning On Ventilation

The ventilation system must be started first before the main power on the continuous unit can be switched on. Referring to Figure 3 the location of the fan starters is shown. Ensure the disconnect on all three starters is in the "ON" position. Push the start button marked "Exhaust" on the operators console to start all three fans. If for any reason one of the fans does not start the rest of the start-up procedure cannot be carried out. Each fan has a differential pressure switch which has to close (ie air flow present) before the main contactor can be closed to power the continuous coal unit.

b) Turning On Main Power

Check that all breakers on the breaker panel (See Figure 3) are in the "OFF" position. Check that the "Level" switch and "Gas" switch on the operators console are both in the "OFF" position.

Push the "Main Power" switch on. The green indicator light above the switch should come on. If it does not come on, there are electrical problems and the electrican should be notified.

c) Turning On Instrument Power

The instrument power is turned on by means of breaker #3 in the breaker panel. This energizes the Combustible Gas Detector and Gas On-Off switch and Level On-Off switch.

The Combustible Gas Detector has a small switch and an audible alarm on its front panel. The switch is labeled "Alarm Silence On-Off". Ensure the switch is in the "On" position and the indicator light next to the switch is off. The gas detector should be tested weekly using a calibration gas set at 50% L.E.L. of methane. The alarm point should trip at 20% L.E.L. of methane.

d) Turning On Residium Feed Heat

The feed tanks #1 and #2 should be charged with residium. Tank #1 heater is turned on with breaker #7 and Tank #2 heater is turned on with breaker #9.

Set the temperature controllers labelled Feed Tank 1 and Feed Tank 2 for the desired holding temperature (about 150°C). Turn on breaker #11 to energise the Heat Trace Temperature Controller which controls heat to the residium feed valves and piping.

e) Starting The Gas Heater

There are a number of valves which have to be open in order to light the pilot light on the gas burner. There is a valve which feeds natural gas to the basement area. This valve is lcoated above the plumbers bench and is normally on. There is a ceiling mounted valve above the bvurner location with a rope attached for activation. This valve is for emergency shutoff and can be closed by pulling on the rope. There is a flexible gas hose extending down from the ceiling to the burner where there is another shutoff valve. This valve has to be open in order to light the pilot as does the stop cock in the 1/4"^{D} tube which extends to the pilot light. A final gas valve just before the burner should be left in the closed position.

The pilot light can now be lit. This is done by holding the red button down on the top of the automatic shut-off valve and lighting the pilot.

The button should be held down for about one minute and the pilot should stay lit when the button is released.

The heat duct should be positioned in the center of the rotary conveyor and adjusted so that it does not catch on the rotating screw. To ensure that the adjustment is proper turn the speed control on the rotary screw drive control box to zero and turn on breaker #5. Turn the speed control up until the rotating conveyor rotates slowly. Make sure the rotating screw does not catch or push the heater hose out of the center of the conveyor. Turn the speed control to zero.

Now the main gas burner can be started. Open the final gas valve at the burner. Turn the "Gas On" swtich to the on posiiton. There is a one minute timer that delays ignition of the burner until the rotating conveyor has been purged. The burner should then ignite.

f) Start-Up Of The Level Control

The level control system utilizes bottled nitrogen supply to feed the bubbler system. This prevents oxidation and helps to prevent blockage of the dip tube in the residium. Ensure the cylinder pressure is above 500 psi to prevent running out of nitrogen during a test run. Ensure the nitrogen regulator is backed out and that the downstream shutoff valve is closed ahead of the low pressure regulator. (See Figure 2). With the high pressure regulator properly tightened onto the nitrogen bottle and the regulator control backed out to provide zero delivery pressure, open the cylinder valve. Then adjust the regulator to give a delivery pressure of 20 PSIG. The valve to the low pressure regulator can now be opened.

NOTE: Do not exceed 200 PSIG on the low pressure regulator input.

The low pressure regulator has been preset at 20 inches water pressure delivery to the supply restriction. The bottom bubbler glass on the control panel should indicate nitrogen flow. The top bubbler should not show a flow. If it does show a flow it indicates that the dip tube in the residium is plugged.

The preheater can now be turned on. The temperature control can be set between 200°C and 1200°C. The proper setting is a function

of the flash point of the residium and should have been previously determined. When the preheater reaches the set temperature residium can be introduced to the rotary conveyor. The level control can now be activated. Ensure that supply air to the valve actuator is at least 50 PSIG and then turn the "Residium On-Off" switch to the "On" position.

The level of residium in the rotating vessel should be observed and switched off when the level approaches one inch from overflow. Wait for one minute then switch the level control on again. The automatic level control should now operate without any overshoot. The level indicator light on the control panel lights when the control valve is open. A change in level of about 0.1 inch will cause the valve to actuate. The end of the dip tube should be between 0.3 and 0.5 inches below the surface of the residium.

g) Turning On The Radiant Heaters

The radiant heaters can be turned on to assist in maintaining the residium temperature at the desired setpoint by switching on breaker #10.

A sense thermocouple protrudes 3/8" beyond the end of the level dip tube and is used in conjunction with the Radiant Heater Controller to control the residium temperature.

APPENDIX 3

Supply and Services Canada | Approvisionnements et Services Canada

11 LAURIER ST., HULL, QUEBEC K1A 0S9
11, RUE LAURIER, HULL (QUÉBEC) K1A 0S9

CONTRACT AMENDMENT
MODIFICATION DE CONTRAT

31ES2

Gulf Canada Limited
2489 Sheridan Way
Sheridan Park, Ontario
L5K 1A8

P.A. - L.P. | Page 1 of 1

DSS FILE NO. - N° DU DOSSIER MAS: 23SQ.23440-4-9062

ADDRESS ENQUIRIES TO: / ADRESSER LES DEMANDES DE RENSEIGNEMENTS À:
Mr. E.K. Fresque (23SQ)
TEL. (819) 997-4214

THE REFERENCED CONTRACT IS HEREBY AMENDED AS FOLLOWS - CE CONTRAT EST PAR LES PRÉSENTES MODIFIÉ COMME SUIT

AMENDMENT NO. / N° DE LA MODIFICATION	DATE	TYPE OF AMENDMENT - GENRE DE MODIFICATION	AMENDMENT VALUE / VALEUR DE LA MODIFICATION	REVISED TOTAL EXPENDITURE / MONTANT GLOBAL REVISÉ
1	AUG / AOUT 27 1985	☐ INCREASE / AUGMENTATION ☐ DECREASE / DIMINUTION ☒ NO CHANGE / AUCUN CHANGEMENT	nil	$279,439.00

TITLE

BENEFICIATION OF LOW RANK CANADIAN COALS WITH HEAVY RESIDUA (PHASE II)
(Part of Energy, Mines and Resources 50/50 Cost Shared Energy Conversion Program)

On page 1 of the contract, at block entitled "DELIVERY"

DELETE: July 31, 1985

SUBSTITUTE: January 31, 1986

On page 2 of the contract, at clause entitled "DELIVERABLE ITEMS"

DELETE: July 31, 1985

SUBSTITUTE: January 31, 1986

On page 3 of the contract, at clause entitled "SCHEDULE OF DELIVERY"

DELETE: July 10, 1985

SUBSTITUTE: January 10, 1986

DELETE: July 31, 1985

SUBSTITUTE: January 31, 1986

At Appendix "A" of the contract, "STATEMENT OF WORK"

ADD: Appendix "A.1" attached hereto

ALL OTHER TERMS AND CONDITIONS OF THE CONTRACT REMAIN UNCHANGED
TOUTES LES AUTRES MODALITÉS ET CONDITIONS DU CONTRAT DEMEURENT INCHANGÉES

REQUISITIONING DEPT. - MINISTÈRE DEMANDEUR	INSPECTION	C. OF PY - C. DE V.A.E.	VALUE OF PY / VALEUR DE V.A.E.
Energy, Mines and Resources 23	☒ CONSIGNEE'S PREMISES / CONSIGNATAIRE ☐ DSS PWU / MAS DMP ☐ DND QAR / MDN QAR	CDN	

All invoices, shipping bills and packing slips must show the following numbers
Les factures, connaissements et fiches d'emballages doivent porter les numéros suivants

REQUISITION NO. AND FINANCIAL CODES - N° DE LA DEMANDE ET CODES FINANCIERS
RN: 23440-4-9062
FC: 234-131-000000-505102-0415

CONTRACT SERIAL NUMBER / NUMÉRO DE SÉRIE DU CONTRAT: 0SQ84-00097

GST/PST - TPS/TVP	VSP - LAP	DSSA - MAS	CONTRACT DATE - DATE DU CONTRAT	APPROVAL AUTHORITY / POUVOIR D'APPROBATION
nil	L5K	Z-0001-N-R-D	July 20, 1984	SSPO

DSS-MAS 10808 (SED)

FOR THE DEPARTMENT / POUR LE MINISTÈRE
E.K. Fresque
SIGNATURE

Canada

APPENDIX "A.1"

MODIFICATION TO THE STATEMENT OF WORK

The Statement of Work, Appendix "A", is to be amended as follows:

The MK-II reactor will be used to study the effect of coal particle size on beneficiation of Wabumun and Poplar River coals. Instead of the scheduled 48 experiments only approximately 15 runs are anticipated. The commissioning of this new reactor will also be included in these tasks.

Products from beneficiation will be subjected to proximate analysis, residuum content, sulfur content, and calorific value determinations. Certain selected samples will also be subjected to ultimate analysis and Hardgrove Grindability Index determination.

Attempts will also be made to investigate the possibility of using this unit to generate data of design importance such as heat requirement, coal fines production, and changes of residuum qualities.

Tasks 5 and 6 - Batch Experimental Program

The failure of the rotary kiln reactor designed and built for this program has significantly delayed the progress of this research program. In order to accomplish the main objective of this program, scope of the work has to be redefined and priority reset for the future work.

The data collected so far in this project suggest that the evaluation of the stirred tank reactor and the vibrating screen should be continued and be given top priority. The batch experimental program, which was not investigated for large coal particles, should also be carried out but on a reduced basis.

Review of economics can probably be deleted to give more attention to the other activities, without great sacrifice of the project objectives.

1. Because the single-stage rotary kiln reactor, designed and constructed for this program, showed poor residuum drainage and heat transfer characteristics it should be substituted by the more reliable two-stage system. This continuous laboratory unit consists of a stirred tank for treating the coal with residuum followed by a vibrating screen for separating the excess residuum from the coal.

2. In order to accommodate the completion of the key components and objective of the contract (i.e. development and operation of the continuous beneficiation process) within the remaining, allocated resources, the following changes will be made:

   (a) reduction of the number of batch experiments from 48 to 15 (Task No. 5)
   (b) reduction of the scope of the review of economics (Task No. 11)
   (c) deletion of a formal presentation at EMR

5. PROPOSAL FOR REVISED WORK STATEMENT

The failure of the rotary kiln reactor designed and built for this program has significantly delayed the progress of this research program. In order to accomplish the main objective of this program, scope of the work has to be redefined and priority reset for the future work.

The data collected so far in this project suggest that the evaluation of the stirred tank reactor and the vibrating screen should be continued and be given top priority. The batch experimental program, which was not investigated for large coal particles, should also be carried out but on a reduced basis.

Review of economics can probably be deleted to give more attention to the other activities, without great sacrifice of the project objectives.

A revised Work Statement and Cost are proposed below and an extension of 6 months is required to complete this research program. The letter R is used to denote a revision and the revised tasks will replace the original tasks in the research contract.

Tasks 5R and 6R ($25,000) - Batch Experimental Program

The MK-II reactor will be used to study the effect of coal particle size on beneficiation of Wabumun and Poplar River coals. Instead of the scheduled 48 experiments only approximately 20 runs are anticipated. The commissioning of this new reactor will also be included in these tasks.

Products from beneficiation will be subjected to proximate analysis, residuum content, sulfur content, and calorific value determinations. Certain selected samples will also be subjected to ultimate analysis and Hardgrove Grindability Index determination.

Attempts will also be made to investigate the possibility of using this unit to generate data of design importance such as heat requirement, coal fines production, and changes of residuum qualities.

Tasks 7R and 8R (120,000) - Design and Construction of Continuous Unit

The revised cost for the construction of the continuous laboratory unit is $120,000. This task has been completed.

Tasks 9R and 10R ($60,000) - Operation of Continuous Unit

Evaluation of the stirred tank reactor and vibrating screen separation will be continued with the objective to process both the Wabumun and Poplar River coal in a continuous operation. The following activities are recommended:

- A study of the operation of stirred tank reactor (heat transfer and agitation required) and changes in properties of coal and residuum.
- Performance evaluation of vibrating screens in controlling final residuum content of the product.
- Laboratory scale continuous operation using Wabumun and Poplar River coals (one combined size fractions for each sample) to generate approximately 25 kg sample each.
- Product evaluations for proximate, ultimate analyses, heating value, residuum content determinations, equilibrium moisture, porosity and tendency for spontaneous combustion.

Task 11R ($5,000) - Review of Economics

The scope covered in this review will be capital cost estimates, determination of operating costs, and an estimate or return on investment.

Task 12R ($5,000) - Preparation of Final Report

A final report will be prepared containing all the data and interpretation of data.

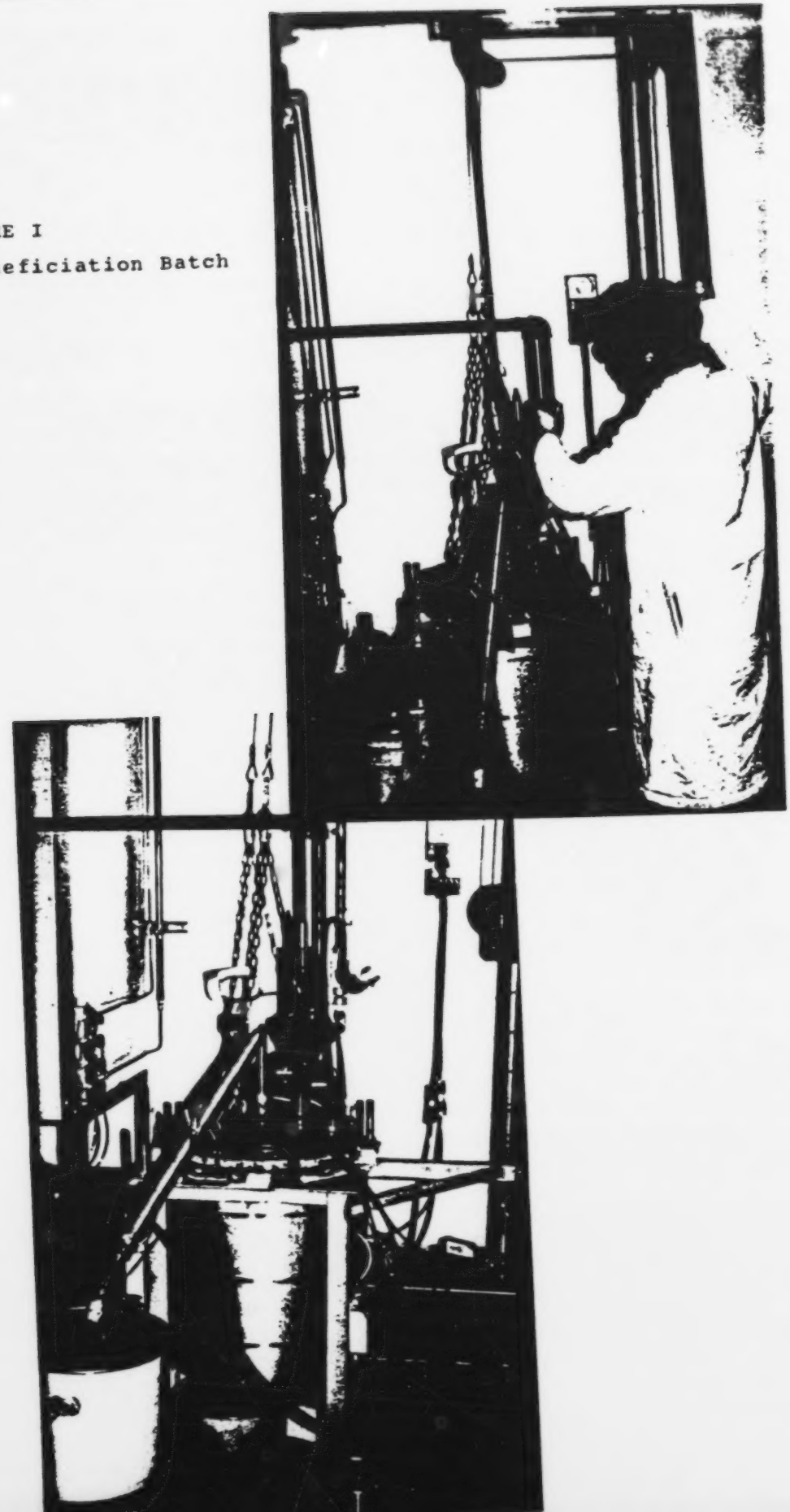
Task 13R - Presentation

Deleted.

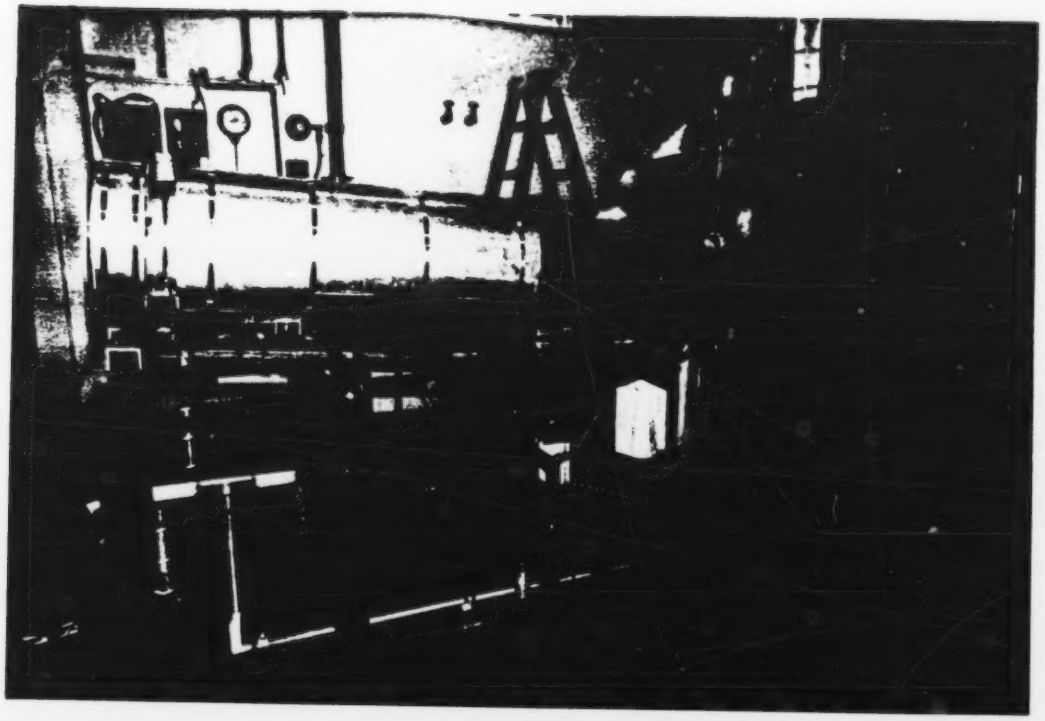
APPENDIX 4

FIGURE I

Coal Beneficiation Batch Unit



APPENDIX 5



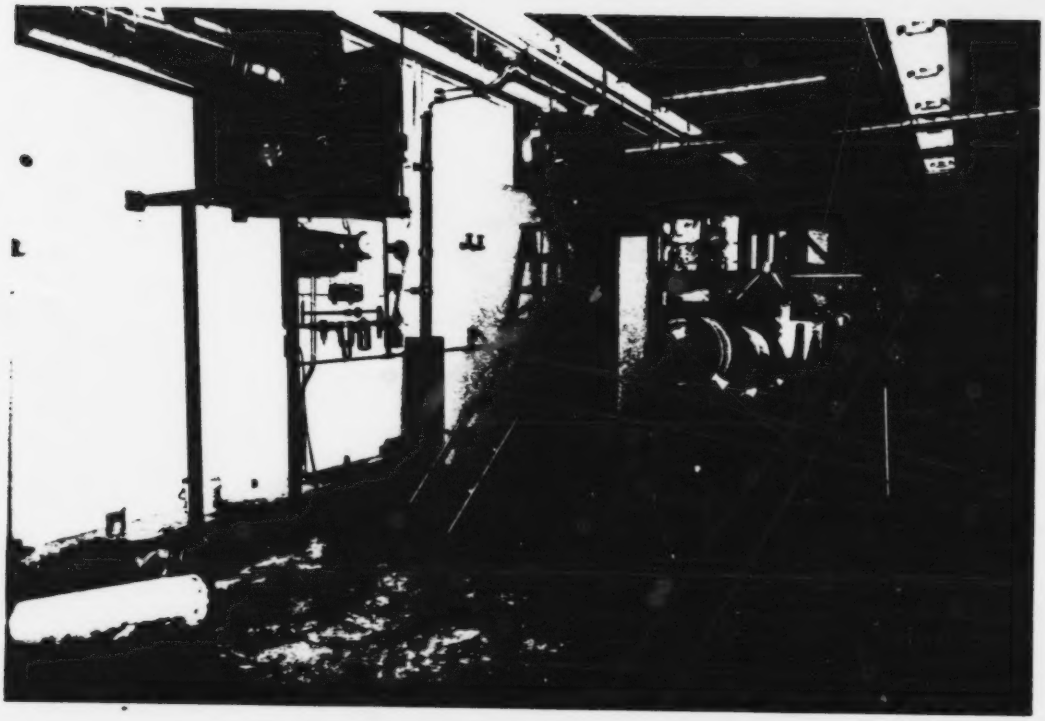


Figure 2(a): Photographs of Continuous Laboratory Unit

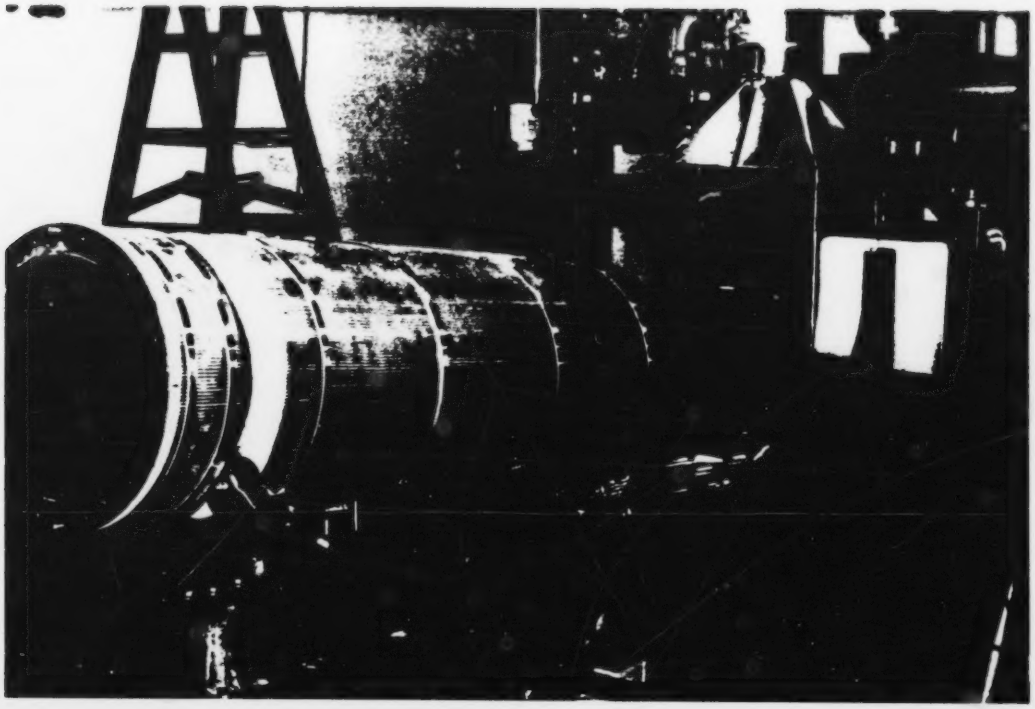


Figure 2(b): Photographs of Continuous Laboratory Unit

Figure 2(c): Photographs of Continuous Laboratory Unit

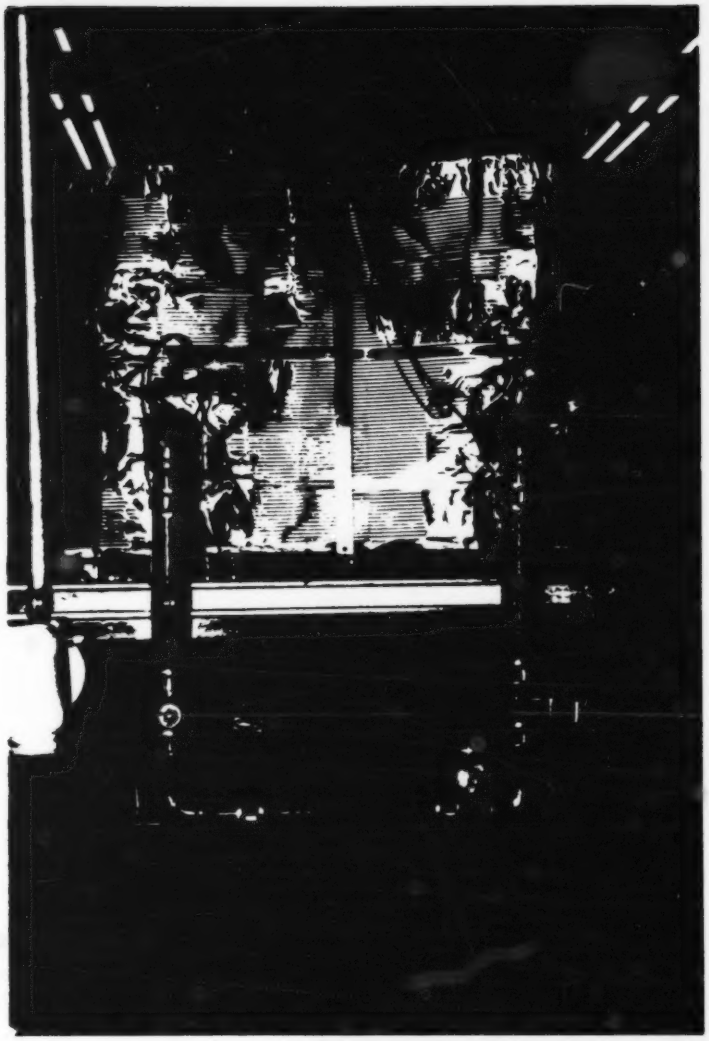
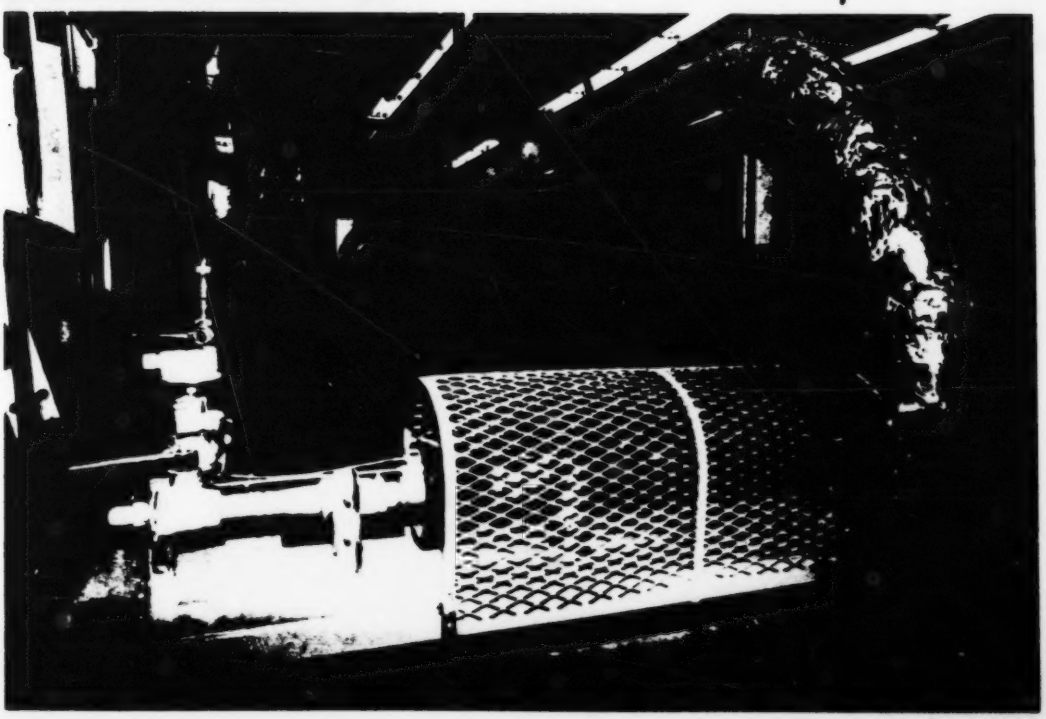


Figure 2(d): Photographs of Continuous Laboratory Unit

Residuum Tanks

Gas Burner.

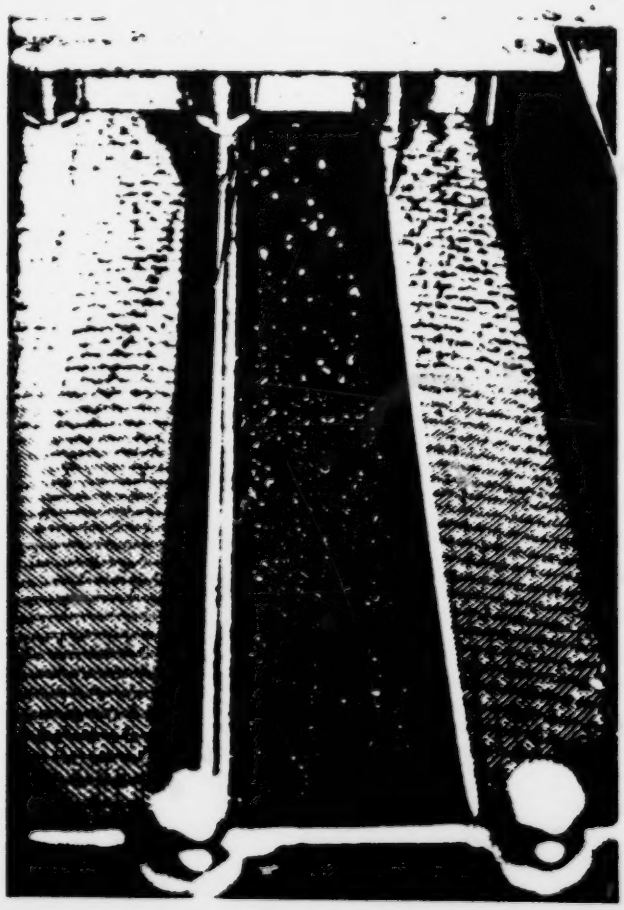


APPENDIX 6

FIGURE 11.

Coal Beneficiation
Continuous Unit

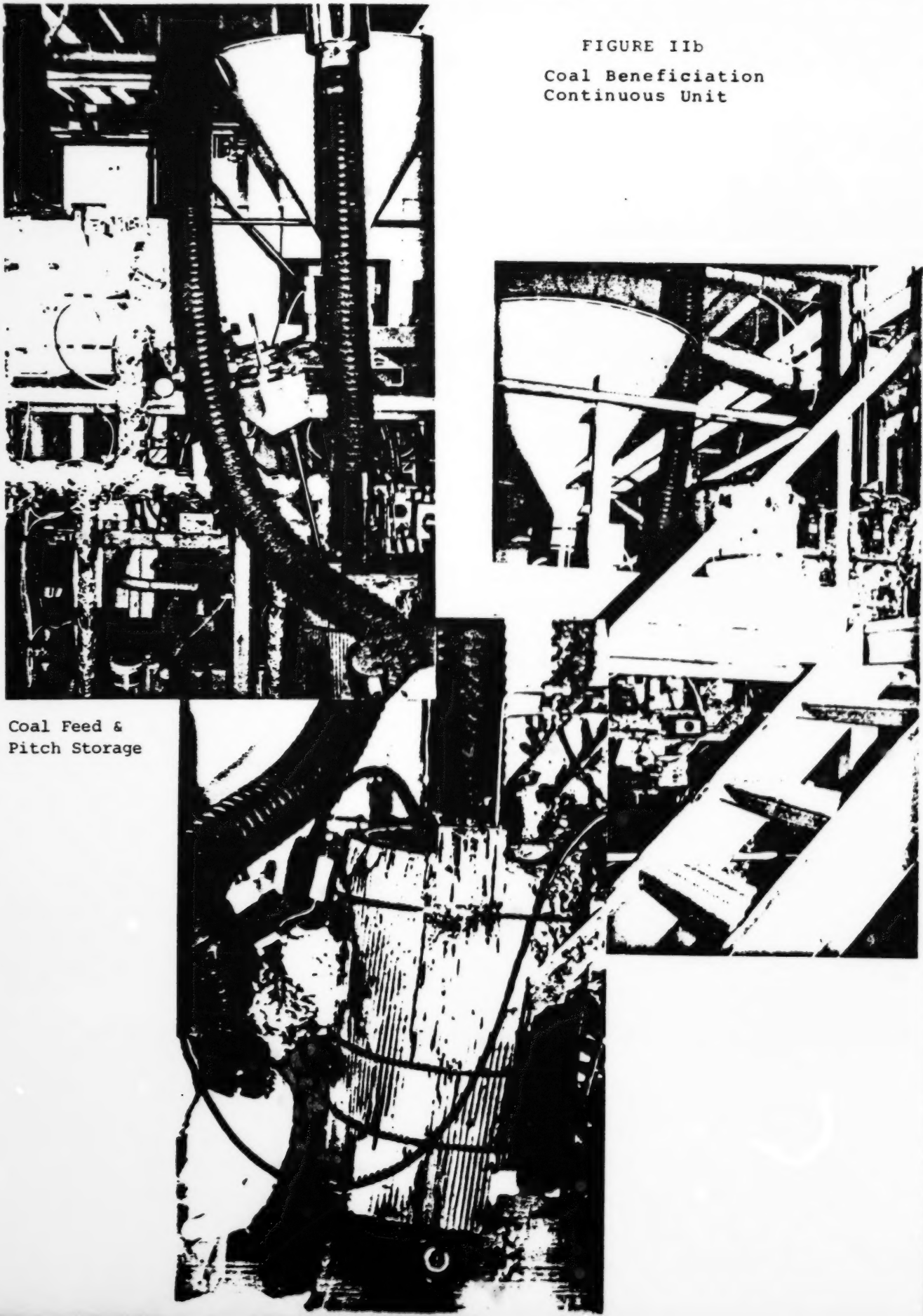
Cover Removal
from Top of
Derrick Screen



View of 4" Wide
Path of Screen

FIGURE IIb

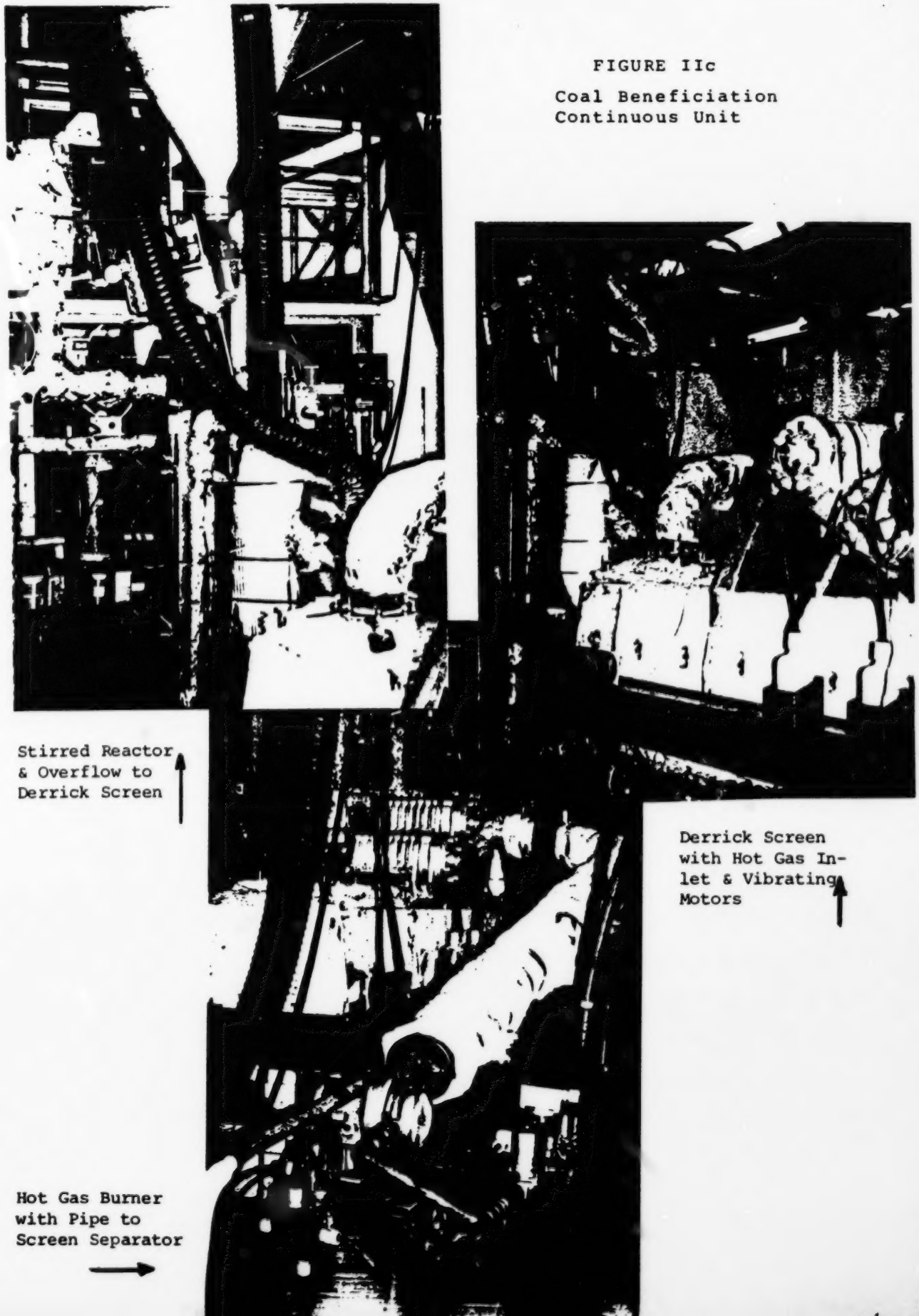
Coal Beneficiation
Continuous Unit



Coal Feed &
Pitch Storage

FIGURE IIc

Coal Beneficiation
Continuous Unit



Stirred Reactor & Overflow to Derrick Screen

Derrick Screen with Hot Gas Inlet & Vibrating Motors

Hot Gas Burner with Pipe to Screen Separator

APPENDIX

NOT

FILMED